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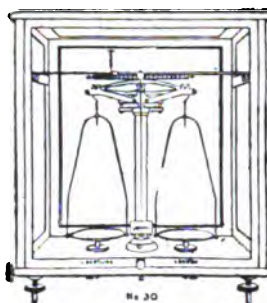
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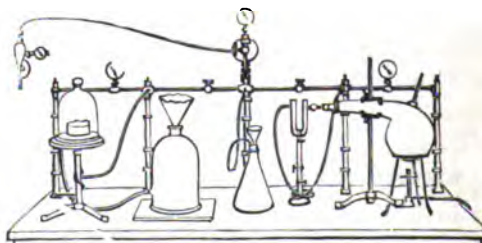
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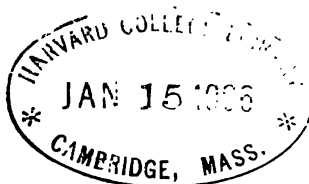
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VOLUME XCIII.

EDITED BY SIR WILLIAM CROOKES, F.R.S., &c.

No. 2406.—JANUARY 5, 1906.

THE PHYSICAL AND CHEMICAL PROPERTIES OF IRON CARBONYL.*

By Sir JAMES DEWAR, M.A., Sc.D., LL.D., F.R.S.,
Jacksonian Professor in the University of Cambridge,

and
HUMPHREY OWEN JONES, M.A., D.Sc., Fellow of Clare
College, and Jacksonian Demonstrator in the University of Cambridge.

THIS paper contains an account, as promised, of a study of the physical and chemical properties of iron carbonyl, similar to that already communicated to the Society, on the properties of the analogous compound of nickel, to which this forms the sequel (*Proc. Roy. Soc.*, 1903, vol. lxxi., p. 427).

The combination of iron and carbon monoxide was discovered by Drs. Mond and Quincke in 1891 (*Trans. Chem. Soc.*, 1891, vol. lix., p. 604), and the resulting compound called iron pentacarbonyl was isolated (as a coloured liquid), and examined by Drs. Mond and Langer in the course of the same year (*Trans. Chem. Soc.*, vol. lix., p. 1090).

Our knowledge of this remarkable substance is derived entirely from the observations of the last-named investigators, and a few isolated observations of others—thus the late Dr. Gladstone determined its refractive indices and Dr. Perkin the magnetic rotation. The molecular refractive power and magnetic rotation are abnormally high, and the compound is diamagnetic; such remarkable properties naturally aroused considerable interest, but the lack of any further work on the compound is doubtless due to the great difficulty experienced in preparing quantities of the compound large enough to work with.

The peculiarities exhibited by this compound call for further attention, and we have examined the formation of the compound and its properties, both physical and chemical; the investigation has been carried out on the same lines as that on nickel carbonyl (*loc. cit.*, and *Trans. Chem. Soc.*, 1904, vol. lxxxv., pp. 203, 213), and attention has been directed more particularly to the differences between the iron and nickel carbonyls, such as the difference in formula, colour, stability, and, more especially, to the action of light on iron carbonyl.

We are indebted to Dr. Mond for two specimens of iron carbonyl with which the investigation was begun; latterly we have been able to prepare and use much larger quantities of the compound, and further work is still in progress. The preparation of iron carbonyl in quantity is a long and tedious process; the yield obtained depends on a number of circumstances; the best conditions for the preparation of the compound are being investigated, and it is hoped that the observations made on this subject will form the subject of another communication.

Liquid iron carbonyl is described as a yellow liquid, with the formula $\text{Fe}(\text{CO})_5$, boiling at 102.8°C . It is remarkable that nickel carbonyl, the compound of a metal whose salts are highly coloured, is colourless, while iron carbonyl is coloured, and the salts of iron have usually only a feeble colour. Also the difference in composition, coupled with the higher boiling-point of the iron compound, caused by the introduction of more carbon monoxide, is a striking phenomenon. We consider it important to determine whether careful purification and drying of the compound would remove the colour, and to procure further analytical evidence bearing on its formula.

To purify the substance it was placed with some suitable drying agent (copper sulphate, barium oxide, zinc chloride, and pure phosphorous pentoxide were all found suitable) in one limb of a carefully-dried glass tube bent into the form of a V-tube; the other limb was empty. The tube was then exhausted by means of a side tube, filled several times with carbon monoxide, hydrogen, or nitrogen, exhausted again, and then the side tube was sealed off. After standing in the dark for some time the iron carbonyl was distilled into the empty limb by placing this in a freezing mixture and the other limb in a beaker of water at about 50°C . The liquid was still of a pale yellow colour, even after standing several days over phosphorous pentoxide and distilling by feeble gas-light.

Another form of experiment showed conclusively that the colour is a definite property, and is not due to impurities. A tube bent into the form of an M was used, the liquid was allowed to stand in one limb for several days over anhydrous copper sulphate, distilled over into the bend, where it was allowed to stand for a week over pure phosphorous pentoxide, and then finally distilled into the empty limb of the tube. The operations were carried out in the dark, and the liquid still possessed its yellow colour. Consequently, it is concluded that the pure compound has a yellow colour. We have therefore not been able to confirm the opinion expressed by Armstrong that the pure compound would be colourless (*Proc. Chem. Soc.*, 1893, p. 58).

For analysis the compound was purified as described above, and rapidly transferred to weighed glass bulbs in a vacuum desiccator, the bulbs sealed and weighed. The transference must be effected very rapidly, as the compound is exceedingly sensitive to air and moisture; a reddish precipitate is produced on standing if the liquid has been exposed even for a very short time to moist air.

The percentage of carbon monoxide was determined by combustion. It was found necessary, in order to get constant results, to pass a current of oxygen through the tube for a long time, while heated to a bright red heat, in order to completely oxidise the carbon which was deposited with the iron.

* A Paper read before the Royal Society, November 16, 1905.

1. 0.2680 grm. of the liquid gave 0.3000 grm. of carbon dioxide.
2. 0.4336 grm. of the liquid gave 0.4825 grm. of carbon dioxide.

The estimation of the iron offered greater difficulties. Mond and Langer (*loc. cit.*) decomposed the compound by heating to 100° C. with chlorine or bromine water in a sealed tube, and then weighing the iron as ferric oxide.

This method always gave slightly high results, and accuracy is limited because small quantities only can be used on account of the great pressure developed.

3. 0.3417 grm. gave 0.1429 grm. of ferric oxide.

A bulb containing a weighed quantity of the liquid was broken under a chloroform solution of bromine, the gas evolved was passed through more of the same solution in a U-tube. When the reaction had ceased the solutions were evaporated to dryness, and the iron precipitated and weighed as ferric oxide.

4. 0.7820 grm. gave 0.3209 grm. ferric oxide.

The best method of decomposing the compound was found to be treatment with alcoholic potash solution in a sealed tube at 100° C. When a bulb containing pure iron carbonyl was broken in alcoholic potash solution, beautiful colourless tabular crystals having a pearly lustre were at once produced; at the surface of the liquid these were at once oxidised by the air with the formation of a reddish brown precipitate.

The mixture contained in a sealed tube was heated to 100° C. for a short time, the tube cooled, opened (no pressure is produced by this decomposition), the contents washed out, and evaporated to dryness. The residue was treated with nitric acid, again evaporated down, and the iron precipitated by hot caustic potash to separate it from aluminium which was dissolved out of the glass.

5. 0.9465 grm. gave 0.3886 grm. ferric oxide,

Several other methods of analysis were also tried, such as decomposing the carbonyl by heat, weighing the iron, and measuring the carbon monoxide. A small bulb containing a weighed quantity of the purified iron carbonyl was placed inside a strong glass bulb, a glass tap was then sealed on to this, the bulb exhausted with a Töpler pump, the small bulb was broken, and the carbonyl decomposed by heating to about 180° to 200° C. until no deposit of iron could be produced by heating a clear portion of the large bulb with a blowpipe flame.

The carbon monoxide was then removed, by means of a Töpler pump, and measured; the bulb with the deposited iron was weighed, the iron dissolved off, and the bulb weighed again after drying. In this process it was found that the glass was often attacked where the iron had been deposited, and the high values obtained for the iron are doubtless to be attributed to this cause and to the presence of carbon in the iron (see later). The following example illustrates the kind of result obtained by this method:—

6. 0.4626 grm. gave 287 c.c. of carbon monoxide at 19° C. and 757 m.m., and left 0.1371 grm. of iron.

	Fe.	CO.	CO by volume for 1 grm.
Found—			
1	—	71.2	—
2	—	70.9	—
3	29.2	—	—
4	28.7	—	—
5	28.7	—	—
6	29.6	—	564.2 c.c. at 0° C. of 760 m.m.

Calculated for—

Fe(CO) ₅	28.57	71.43	571.4 c.c.
Fe(CO) ₄	33.33	66.66	530.9 c.c.

The compound is therefore Fe(CO)₅.

Attempts were made to determine the ratio Fe:CO in the compound by passing nitrogen through a weighed or unweighed quantity of pure iron carbonyl, and then

through a glass tube bent twice on itself, and heated in a bath so that the vapour was heated for some time, and decomposed into iron and carbon monoxide; the iron was deposited in the tube, and then weighed, the carbon monoxide was passed over hot copper oxide and weighed as carbon dioxide in a potash tube. It had been found previously that this process gave good results in the case of nickel carbonyl.

With iron carbonyl, however, consistent results could never be obtained. In the first place the compound is much more difficult to decompose completely than nickel carbonyl, and it was found that, in order to ensure complete decomposition, it was necessary, when keeping the bath at 180° to 200° C., to heat a small portion of the tube nearer the copper oxide tube nearly to redness. Even then it was always found that the sum of the weights of the iron and carbon monoxide was always less than that of the iron carbonyl taken. This was traced to the fact that the iron deposited was always contaminated with carbon, which was given off as hydrocarbons when the iron was dissolved in acids; also the weight of ferric oxide obtained from the iron was always less than it should have been. The ratio CO:Fe was usually about 4.5 to 4.8; this is readily accounted for by a small percentage of carbon in the iron. The presence of carbon in the iron would also tend to raise the percentage of iron as estimated by Method 6.

Further evidence in support of the formula Fe(CO)₅ was afforded by experiments on the decomposition of the vapour enclosed in a space at a given pressure and measuring the pressure of the carbon monoxide produced; this was approximately five times the original pressure of the vapour. These experiments will be referred to later.

Molecular Weight.—Numerous vapour-density determinations have been made, and will be described in another part of this paper. When no dissociation occurs the vapour density varied between 98 and 100, so that the molecular weight corresponds to the formula Fe(CO)₅ with a molecular weight of 198.

Two determinations of the molecular weight in benzene were made by the cryoscopic method; a weighed quantity of liquid in a bulb was broken under the benzene. Owing to the very great sensitiveness of the compound air and moisture, the solution always began to turn turbid before the end of the experiment, even when the greatest care was taken to exclude moisture and the air in the apparatus had been replaced as completely as possible by nitrogen or carbon monoxide.

0.2956 grm. in 14.28 grms. benzene gave a depression of 0.524° C. Hence the molecular weight is 197.

0.3900 grm. in 14.20 grms. benzene gave a depression of 0.705° C. Hence the molecular weight is 194.

Specific Gravity of the Liquid.—Mond and Langer (*loc. cit.*) gave the density of liquid iron carbonyl as 1.4664 at 18° C. compared to water at the same temperatures, or 1.4688 compared to water at 4° C. Gladstone (*Phil. Mag.*, 1893, [5], vol. xxxv., p. 204) gave the density at 13.4° C. as 1.474, at 15.5° C. as 1.470, and at 22° C. as 1.460.

The specific gravity was determined with the aid of a small glass pycnometer (volume 2.2 c.c.) at temperatures from 0° C. to 80° C. Great care was exercised to exclude moisture, and the experiments were carried out in a dim light; a very small amount of decomposition, indicated by the formation of minute bubbles of gas, always took place, which was more marked at the higher temperatures, and was doubtless caused by traces of moisture and air, so that the numbers obtained can only be regarded as approximate for the two highest temperatures. The specific gravity is in each case referred to water at 4° C.

Temperature.	Specific gravity.
0.0° C.	1.494
16.5° C.	1.468
40.0° C.	1.421
61.5° C.	1.382
80.0° C.	1.351

These specific gravity determinations were made with a specimen of the carbonyl kindly supplied by Dr. Mond. When a much larger quantity was available this was purified, and further determinations were made with a much larger pycnometer (volume 7.1100 c.c.), using the same precautions (in this case there were no signs of any decomposition).

Temperature.	Specific gravity.
0° C.	1.4937
21.1° C.	1.4565
40.0° C.	1.4330
60.0° C.	1.3825

These results agree fairly well with those given above, but are naturally most trustworthy on account of the larger quantity of material used.

The coefficient of expansion for 0° C. to 21° C. is 0.00121, for 21° C. to 40° C. is 0.00128, and for 40° C. to 60° C. is 0.00142. The mean coefficient of expansion is therefore about 0.00138.

By extrapolation on the curve the specific gravity at the boiling-point, 102.5° C., is 1.310. The molecular volume is therefore 149.6. At the melting-point, -20° C., the specific gravity is 1.53 and the volume of the molecule is then about 128.

The values of the specific gravity of the liquid at 0° C. and 60° C., taken with the critical temperature (288° C., for which see *prox.*), give the following Waterston formula for the relation between the volume v and the temperature t° C. :—

$$v = 1.974 - 0.5307 \log (288 - t^{\circ}).$$

Refractive Indices.—Gladstone (*loc. cit.*) determined the refractive indices of the carbonyl for several of the lines of the spectrum, and found that, as in the case of nickel carbonyl, the compound had a very large molecular refractive power and an enormous dispersive power. It was therefore unnecessary to investigate this property minutely, and two determinations only were made for sodium and thallium light with an Abbé refractometer by Mr. A. Hutchinson, of the Mineralogical Department.

μ for Na light	1.519
μ " Tl "	1.528 at 22° C.

These values agree very closely with those found by Gladstone—1.5180 and 1.5289.

Melting-point.—The pale yellow liquid crystallises when cooled to a pale yellow solid which melts at -19.5° C. to -20° C. At the temperature of liquid air the solid entirely loses its colour, which it gradually recovers on warming up again.

Vapour Pressure and Boiling-point.—The vapour pressure was determined by the static method as in the case of nickel carbonyl.

A wide barometer tube, drawn off to a fine capillary at one end, was carefully cleaned and placed upright in a vessel of pure dry mercury, in a room lighted by a feeble gas-jet. A small tube containing iron carbonyl was now introduced, the whole exhausted thoroughly by means of a Fleuss pump, and sealed off at the end. The pressure was then read off by means of a kathetometer, while the tube was surrounded by a bath at various temperatures. Observations taken after cooling and allowing the tube to stand showed that no appreciable amount of decomposition took place under the conditions of the experiment. The results are appended below :—

Temperature.	Pressure.
-7.0° C.	14.0 m.m.
0.0° C.	16.0 "
16.1° C.	25.9 "
18.4° C.	28.2 "
35.0° C.	52.0 "
57.0° C.	133.0 "
78.0° C.	311.2 "

Next day at 18.9° C. the pressure was 29.4 m.m.

The boiling-point given by Mond and Langer is 102.8° C. at 749 m.m. Several determinations of the boiling-point were made, all of which gave a result slightly lower than this. Thus, it was found that the liquid boiled at 101.8° C. at 736 m.m., at 102.0° C. at 744 m.m., and at 102.7° C. at 764 m.m. The values for 0° C. and 102.7° C. give the following Rankine formula for the relation between the vapour pressure p in m.m. of mercury and the absolute temperature T :— $\log p = 7.349 - 1681/T$. This fits in very well with the results for the intermediate temperatures.

Critical Temperature.—It was found by trial experiments that the liquid could be heated in a glass tube under pressure to temperatures considerably above its boiling-point without undergoing noticeable decomposition. Its critical temperature was therefore determined by placing some liquid in small thick-walled capillary tubes, exhausting, sealing-off, and then heating the tubes in an air- or paraffin-bath until the meniscus disappeared. The tubes were about one-third to one-half full of liquid, and though no deposit of iron was produced, yet the tubes burst after heating several times to the critical point. In several determinations it was found that the meniscus disappeared between 285° C. and 288° C.

The formula $T = 0.66 T_c$, where T_c is the absolute boiling-point and T the absolute critical temperature, is usually applicable to liquids which are not associated in the liquid or gaseous state near the boiling-point, and was found to be applicable to nickel carbonyl; it should therefore be applicable here. Taking the boiling-point as 102.5° C., the critical temperature should be 289.2° C., a value agreeing very closely with the number found experimentally.

The critical density of iron carbonyl is calculated to be 0.49, the value for nickel carbonyl is 0.46. The critical pressure calculated from the Rankine formula for the vapour pressure is 29.6 atmospheres.

The number obtained by dividing the absolute critical temperature by the critical pressure, which is proportional to the volume of the molecule, Van der Waal's constant b , is 18.9, the corresponding value for nickel carbonyl is 15.5, and the value for carbon monoxide is 3.7. Hence the volume of the molecule of iron carbonyl is 5.1 times larger than that of carbon monoxide, while 4.2 represents the ratio for the nickel carbonyl.

The latent heat of iron carbonyl is 39.45 calories per grm., and the Trouton constant, molecular latent heat divided by absolute boiling-point, is 20.6, a value identical with that for nickel carbonyl.

The molecular volume of iron carbonyl at its boiling-point is 150, so that taking 7.0 as the volume of the iron atom, we get 28.6 as the volume of each carbon monoxide molecule, a number smaller than that found for nickel carbonyl, i.e., 32.2. Liquid carbon monoxide at its boiling-point has a molecular volume of 35, so that a greater contraction would take place in the formation of liquid iron carbonyl from liquid carbon monoxide and iron, if that were possible, than in the formation of nickel carbonyl under similar conditions.

The similarity between many of the constants of nickel and iron carbonyls is very striking, in spite of the fact that the substances differ so widely in their boiling-points and stability.

(To be continued).

"Annuaire du Bureau des Longitudes."—The house of Gauthier-Villars, of Paris, has just published the 1906 edition of the above useful annual. In this compact volume are to be found, as usual, a fund of information equally interesting to the engineer, the physicist, and the chemist. We may specially mention a well-illustrated article by G. Bigourdan on Eclipses of the Sun, with instructions as to observations which can be made during an eclipse. The book contains nearly 900 pages, 16mo., and has that rarity in a French book, a good alphabetical index.

THE CANYON DIABLO METEORITE.

Coon Mountain and its Crater.—Dr. Dixon announced that Mr. Daniel Moreau Barringer and Mr. Benjamin Chew Tilghman, members of the Academy, had notified him of their discovery that the crater of Coon Mountain or Coon Butte, in Northern Arizona, twelve miles south-east of Cañon Diablo Station on the Atchison, Topeka, and Santa Fé Railway, is an impact crater and not a crater produced by a steam explosion, as has been supposed since the examination made of it by members of the United States Geological Survey. They have proved, by a large amount of development work, according to their statements, that the large crater and elevation known as Coon Mountain is the result of a collision with the earth of a very large meteorite or possibly a small asteroid, fragments of which are well known to the scientific world by the name of the Cañon Diablo siderites. Their development work, consisting of cuts, shafts, and bore-holes, has established the following facts:—

1. That the formation of the crater and the deposition of the meteoric material were simultaneous.
2. That meteoric material has been found five hundred feet below the surface of the centre of the crater.
3. That sandstone supposed to be in place exists less than one thousand feet below the surface of the centre of the crater.

Mr. Barringer and Mr. Tilghman have presented to the Academy for publication two comprehensive papers in which they set forth in full their reasons for the above statements.—*Proceedings of the Academy of Natural Sciences of Philadelphia*, December 5, 1905.

NEW RESEARCHES ON THE ATOMIC WEIGHT OF NITROGEN.*

By PHILIPPE A. GUYE.

(Continued from vol. xcii., p. 287).

III.

HAVING demonstrated our second thesis, we have to express the results relative to the atomic weight of nitrogen to which the different methods just discussed lead. We will first recall the figures at which the different authors have successively arrived.

Leduc (molecular volumes, 1897)	14.005
Berthelot (density limits, 1898)—	
Starting from N ₂	14.007
" " N ₂ O	14.000
Guye and Friederich (density limits, 1900)—	
Starting from N ₂	14.005
Lord Rayleigh (density limits, 1905)—	
Starting from N ₂	14.008
" " N ₂ O	13.998
Guye (reduction of critical elements, 1905)—	
Starting from N ₂	14.007
" " N ₂ O	14.006
" " NO	14.009
Gray (density limit, 1905)—Starting from NO ..	14.006

The mean of the determinations of 1905, which are the most accurate—excluding the evidently too low value furnished by the density limit of N₂O—is N = 14.007.

The variations between these different determinations are due, in part at least, to the different selections which were made of the different density determinations, in part equally to the fact that one is not always placed under conditions reducing to a minimum the correction for the density of the gas which results from the experiment.

These conditions are best realised as follows:—

When the densities, L and L', of any two gases are compared, the molecular weight, M, found will bear to the known molecular weight, M', a relation of the form—

$$M = M' \frac{L}{L'} \frac{(1 + \lambda)}{1 + \lambda'} = M' \frac{L}{L'} (1 + \epsilon);$$

in which (1 + λ) and (1 + λ') represent the correction factors calculated by one or other of the four methods that we have just described. For brevity, we write—

$$\frac{1 + \lambda}{1 + \lambda'} = 1 + \epsilon.$$

If, instead of comparing all gases to one taken as standard (oxygen), the comparisons are limited to gases taken in two's, so that for each pair of gases the critical constants T_c and p_c are near, or at least of the same order of magnitude, it is evident that the factor (1 + ε) will be very near unity. It is therefore under these conditions that the greatest degree of accuracy is obtained with certainty, and it is of all the greater interest to endeavour to realise them, that the theory of corresponding states, upon which all the physico-chemical methods depend, is not absolutely rigidly expressed as a physical law; this repeats that the physico-chemical calculation of the molecular weight of a gas represents as yet an approximation. It is therefore of great interest to work under conditions giving the greatest degree of accuracy. One is the more forced to consider as exact the corrections deduced from this theory in proportion as the latter are themselves slight.

This method of procedure presents another advantage: it places in evidence the numerical value of the correction factor (1 + ε) obtained by each of the physico-chemical methods, and also estimates the degree of concordance with which it is determined.

The following are the results which are thus obtained with the nitrous gases:—

Corrected Ratio of the Densities of Nitrogen and Oxygen.

—The ratio of the densities of these two gases resulting directly from experimental data at 0° and 1 atmosphere is—

16 L'/L = 14.003 (Rayleigh), 16 L'/L = 14.001 (Leduc).

A mean value may therefore be adopted,—

$$16 L'/L = 14.002.$$

The correction factor determined by physico-chemical methods has the following values, in connection with which are inscribed the atomic weights of nitrogen obtained by correcting the rough value 14.002.

TABLE IX.

Methods.	Correction factor.	N.
1. Density limits at 0°:—		
Berthelot	1.00038	14.008
Rayleigh	1.00038	14.008
2. Reduction to 0° of the critical elements:—Guye	1.00044	14.008
3. Corresponding densities:—		
N ₂ , 37°/503; O ₂ , 100°/760		
(Guye)	1.00085	14.014
Corresponding temperature,		
Avogadro (Guye)	1.00061	14.011
Corresponding molecular vol.		
at 0° (Leduc)	1.0004	14.008
4. Densities at 1067° (Jaquero		
and Perrot)	1.00043	14.008
Mean	1.00050 × 14.002	14.009

Corrected Ratio of the Densities of the Gases N₂ and CO.

—The direct ratio of these densities, calculated from experimental data, putting CO = 28.002, is—

28.002 L/L' = 28.008 (Rayleigh),

28.002 L'/L = 28.006 (Leduc).

* From the *Bulletin de la Société Chimique de Paris*, 1905.

From the agreement of these results, a single mean value may be adopted—

$$28.002 \text{ L/L} = 28.007.$$

The values of the correction factor, determined by different methods, are placed in the following table, together with (1) the resulting molecular weight M of nitrogen; (2) the corresponding atomic weight N:—

TABLE X.

Methods.	Correction factor.	M.	N.
Density limits (Berthelot) . . .	1.00008	28.009	14.005
" (Rayleigh) . . .	1.00025	28.014	14.007
Reduced to 0° of the critical element (Guye) . . .	1.00017	28.012	14.006
Corresponding density: N ₂ , -11°/719; CO ₂ , 0°/760 (Guye)	1.00014	28.011	14.006
Corresponding temperature, Avogadro (Guye) . . .	1.00014	28.011	14.006
Corresponding molecular volume, 0° (Leduc) . . .	1.0001	28.010	14.005
Mean . . .	1.00015	28.011	14.006

Corrected Ratio of the Densities of the Gases N₂O and CO₂.—The direct ratio of the densities, referred to CO₂ = 44.002, that is, R = 44.002 L/L', has given experimentally the following values:—44.040 (Leduc), 44.020 (Rayleigh), 44.017 (Guye and Pintza).

The mean value is—

$$R = 44.025.$$

With the liquefiable gases, I have already indicated that the method of density-limits does not give the same accuracy as with the permanent gases; one can therefore confine oneself to calculating the value of the correction factor by the method of corresponding densities and by that of reduction to 0° of the critical elements, whence we have the following table (M = the corrected molecular weight of N₂O):—

TABLE XI.

Methods.	Correction factor.	M.	N.
Corresponding density: N ₂ O, 0°/760; CO ₂ , -4.5°/739 . . .	1-0.00033	44.011	14.006
Reduction of the critical element . . .	1-0.00023	44.015	14.008
Mean . . .			14.007

The uncertainty ± 0.002 on the atomic weight of carbon is again reduced in this case to ± 0.001 on the atomic weight of nitrogen.

Corrected Ratio of the Densities of the Gases NO and O₂.—Gray has recently pointed out a preliminary value of the density of nitric oxide. From this is deduced for the weight of a normal litre L = 1.3402, which value is confirmed by experiments executed in my laboratory with the co-operation of Davila.

Taking for oxygen L = 1.4290 (mean of all the observations), the direct ratio is—

$$R = 32 \text{ L/L} = 30.012.$$

This value not being definitive, the correction factor has been calculated only by two methods; it is given in the following table, with the molecular weights M of NO, and the atomic weight N deduced from them.

TABLE XII.

Methods.	Correction factor.	M.	N.
Density limit (Jaquero and Schener) . . .	1-0.00020	30.006	14.006
Reduced to 0° of the critical element (Guye) . . .	1-0.00008	30.010	14.010
Mean . . .			14.008

Resumé of the Physico-chemical Values of the Atomic Weight of Nitrogen.—The preceding tables give rise to the following recapitulation of the means:—

Ratio.	N.
N ₂ : O ₂ (6 values) . . .	14.009
N ₂ : CO (6 values) [C = 12.002] . . .	14.006
N ₂ O : CO ₂ (2 values) [C = 12.002] . . .	14.007
NO : O ₂ (2 values) . . .	14.008

General mean . . . 14.008

The general mean of these four ratios of gas densities, corrected by physico-chemical methods, is therefore—

$$N = 14.008,$$

the greatest range being only 0.003.

In consigning this final value, which may be provisionally be rounded into 14.01, I must make the reminder that the method of density-limits has only been applied to the permanent gases.

With this reservation, one can but be struck with the agreement of the results, all the more remarkable that the experimental elements serving for the calculation of the correction factor have generally not been determined by the same experimenters as those to whom are due the values of the densities.

(To be continued).

THE VOLUMETRIC ESTIMATION OF CYANATES.*

By A. C. CUMMING, B.Sc., and ORME MASSON, F.R.S.

THE want of a correct and convenient method for the quantitative analysis of solutions containing carbonate and cyanates led to the trial and adoption of the following convenient volumetric process.

To gravimetrically separate carbonate and cyanate from the solution of, say, the potassium salts, is apparently easy in the absence of cyanides and other complicating ingredients. The obvious method is to precipitate the carbonate with excess of barium nitrate, and then to throw down the cyanate from the filtrate by adding excess of silver nitrate. The BaCO₃ may be weighed as such, or as BaSO₄, and the AgCNO may be weighed on a tared filter, but is best dissolved in dilute nitric acid (thus eliminating any chloride that may be present), and precipitated for weighing as AgCl. In connection with such a separation the following points require notice.

J. W. Mellor (*Zeit. für Anal. Chem.*, xl., 19) stated that barium nitrate cannot be used for precipitating the carbonate from commercial cyanide solutions because barium cyanate is also insoluble, and he recommended the use of calcium nitrate. Others have since repeated the statement, and apparently it is accepted as true. Yet, on the face of it, it is extremely improbable that barium should form an insoluble cyanate; it is contradictory to the accounts of the salt given by Wöhler and Berzelius (see "Watts's Dict.," Old Edition, ii., 193); and we have found it to be, as a matter of fact, quite incorrect, for numerous trials make it certain that Ba(CNO)₂ is not precipitated from cyanate solutions by addition of barium nitrate in the cold. We have, however, observed that when excess of barium hydroxide is employed (precautions being taken to exclude atmospheric CO₂) the clear filtrate from the carbonate slowly deposits a further precipitate; but this precipitate is not cyanate but carbonate produced from it by slow decomposition, probably according to the equation $\text{KCNO} + \text{Ba}(\text{OH})_2 + \text{H}_2\text{O} = \text{BaCO}_3 + \text{KOH} + \text{NH}_3$. Similar decomposition of Ba(CNO)₂ solutions is known to occur on boiling ("Watts's Dict.," *loc. cit.*); and it is probable

* Communicated by the Authors. From the *Proceedings of the Society of Chemical Industry of Victoria*, July—August, 1903.

that Mellor's statement that $\text{Ba}(\text{CNO})_2$ is insoluble is to be traced to this action. We find it quite unnecessary to substitute calcium nitrate for the barium salt, and we prefer the latter.

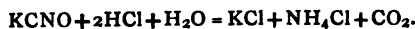
In either case, however, the precipitation of carbonate should be effected without heating, and the addition of ammonia or other alkali must be avoided, as this would lead to difficulties in the subsequent precipitation of silver cyanate. The question occurs, therefore, whether the carbonate precipitation is complete under these conditions. They are not the most favourable, but we believe that it may be taken as nearly, though not quite, complete, provided that a large enough excess of $\text{Ba}(\text{NO}_3)_2$ be used. The following tests were made with an approximately normal Na_2CO_3 solution, using the same quantity and the same total dilution in each case. The amount of reagent used was double the calculated quantity, except in the first precipitation with $\text{Ca}(\text{NO}_3)_2$, where it was somewhat less. A still larger excess should be used in practice, and probably gives more complete precipitation; but it appears that the results tend to be low, though barium nitrate is better than the calcium salt. Titrations of the solution with methyl orange and congo are given for comparison.

Normality of Na_2CO_3 Solution.

By titration { Methyl-orange .. 0.992 } Mean value, 0.990
 { Congo 0.987 }
 By $\text{Ca}(\text{NO}_3)_2$ precipitation—(1) 0.886, (2) 0.937, (3) 0.923
 By $\text{Ba}(\text{NO}_3)_2$ precipitation—(1) 0.963, (2) 0.976

If a cyanate solution contain only normal carbonate, the filtrate from the barium precipitate is exactly neutral, and is in a fit state for the addition of AgNO_3 . If, however, any bicarbonate be present, the precipitation with $\text{Ba}(\text{NO}_3)_2$ is of course incomplete without the addition of ammonia, and it then becomes necessary to exactly neutralise the filtrate. This is troublesome, and apt to introduce errors. Excess of NH_3 would dissolve AgCNO , while any free acid would decompose some of the cyanate; and of course free caustic alkali must be avoided as it would precipitate silver oxide. Another objection to the determination of cyanates as AgCNO is that this precipitate is sometimes contaminated with reduced silver, though this is not always the case. Under favourable conditions, however, it may be taken as giving correct results, as it has been shown by Walker and Hambly (*Trans. Chem. Soc.*, lxvii., 747) that AgCNO is practically insoluble in water containing excess of AgNO_3 , and may be washed with but little loss.

Apart from the faults of the gravimetric process, it is obvious that a volumetric method is preferable where frequent analyses have to be made, provided that a sufficiently accurate one is available. We have found the following method satisfactory so far as it has been yet tested. It depends on the fact that cyanates are, as such, neutral to indicators, but that when boiled for a short time with excess of dilute mineral acid they neutralise a part of it, being quantitatively converted into CO_2 , which escapes, and salts of the metal and ammonia,—



Allen describes ("Commercial Organic Analysis," vol. iii., Part III., p. 484), and others have recommended, an analytical method in which the insoluble AgCNO is similarly used to neutralise standard nitric acid; but, so far as we know, the following process, which depends primarily on the neutrality of soluble cyanates as such, is novel.

It may be briefly described thus:—A known volume of the mixed carbonate and cyanate solution is first titrated in the cold with standard acid, using methyl-orange or congo indicator. It is important to use a neutral solution of the indicator for comparison, and to record the exact point when the first change of tint is observed, for the decomposition of cyanate by excess of acid makes accurate back titration impossible. After thus obtaining the measure of the carbonate, a sufficiently large measured excess of the

acid is run in, when it will be seen that there is (as a consequence of the above reaction) a long range of neutral tint, only gradually deepening to the true acid colour. The mixture is then boiled for a few minutes to ensure the complete decomposition of the cyanate and to expel the CO_2 . The boiling may be stopped when bumping sets in, and the solution is cooled, and more indicator added if necessary. The residual excess of acid is now determined by titration back with standard alkali. If we call this residual excess n^1 , and the original added excess n , it is obvious from the equation for the decomposition of cyanate by acid, that $\frac{1}{2}(n - n^1)$ is the measure of the cyanate.

A second and independent measure of the cyanate may be obtained as follows:—After the last-mentioned titration, a sufficient excess of the alkali is run in, and the mixture is now boiled till all the ammonia has been expelled. The mixture is cooled, given additional indicator if necessary, and titrated back with acid, so as to determine the residual excess of alkali. If we call this residual excess m^1 , and the original added excess of alkali m , $m - m^1$ is the measure of the quantity of NH_3 expelled. This is of course equivalent to the cyanate, unless the original solution contained ammonium salts. In such a case $(m - m^1) - \frac{1}{2}(n - n^1)$ gives a correct measure of their amount. It may be here mentioned that in the methods described by Herting (*Journ. Soc. Chem. Ind.*, Abstracts, xx., p. 838) and Milbauer (*Journ. Soc. Chem.*, Abstracts, 1903, ii., p. 392), for the analysis of commercial cyanide solutions, the cyanate determination is based entirely on the amount of ammonia present after boiling with acid, and that they are therefore necessarily misleading if, as sometimes happens, the cyanide contains NH_4 salts. In working our method we employ decinormal sulphuric acid and soda, and the analysis is done simultaneously in duplicate.

The following tests were made with a solution of potassium carbonate and cyanate, prepared by careful addition of red-lead to fused KCN. The solution contained a trace of KCN, which was determined by titration with AgNO_3 , and allowed for in calculating the results. The gravimetric determinations of carbonate and cyanate, obtained by the methods already discussed, are given for comparison.

Analysis of Solution of K_2CO_3 and KCNO .

(Results stated in grm.-equivalents per litre).

I. Gravimetric Analysis.

Carbonate (by precipitation with barium nitrate):—(1) 0.0178, (2) 0.0151, (3) 0.0165, (4) 0.0168, (5) 0.0177. Mean, 0.0168.

Cyanate (by precipitation with silver nitrate):—(1) 0.0429, (2) 0.0413, (3) 0.0420, (4) 0.0439, (5) 0.0425. Mean, 0.0425.

II. Volumetric Analysis.

Carbonate (titration in the cold):—(1) 0.0182, (2) 0.0181, (3) 0.0181, (4) 0.0182. Mean, 0.0182.

Cyanate (titration after acid boiling):—(1) 0.0440, (2) 0.0430, (3) 0.0429. Mean, 0.433.

Cyanate (titration after alkaline boiling):—(1) 0.0412, (2) 0.0429, (3) 0.0406, (4) 0.0414. Mean, 0.0415.
 Mean of all volumetric results for cyanate, 0.0424.

Obviously, the volumetric method here described has but limited scope, unless it can be used in the analysis of commercial cyanide.

(To be continued).

Sixth International Congress of Applied Chemistry—Rome, 1906.—The Sixth International Congress of Applied Chemistry will be held in Rome in April, 1906, during Easter week. All communications should be addressed to the General Secretary, Prof. Vittorio Villavecchia, Via Panisperna 89, Rome.

THE CHEMICAL SEPARATION OF THE RADIO-ACTIVE TYPES OF MATTER IN THORIUM COMPOUNDS.*

By HERMAN SCHLUNDT and RICHARD B. MOORE.

OUR knowledge of the nature of the radio-activity of thorium is largely due to the extensive researches of Rutherford and Soddy (*Journ. Chem. Soc.*, 1902, lxxxi., 131, 837). These investigators have shown that the radio-activity of the ordinary thorium compounds on the market consists of four parts, due to four specific types of matter. On the basis of their theory of successive disintegration, the parent thorium is slowly but constantly undergoing change. A very small fraction of the thorium atoms is continuously breaking up, giving off α -particles and leaving a new type of matter, Thorium X, which is also radio-active. Thorium X, in disintegrating, gives rise to a radio-active gas, the Emanation, which breaks up rapidly, sending out particles and depositing a solid on near-by objects. This solid likewise emits radiations and is therefore responsible

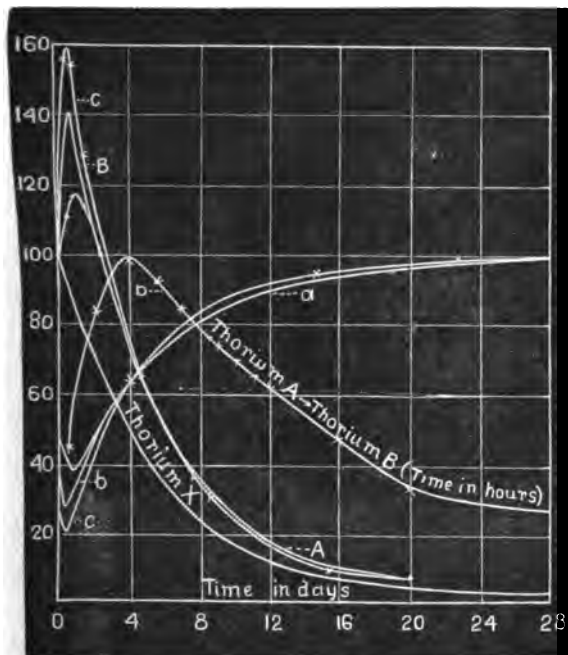


PLATE I.

Ammonia, A a; Pyridine, B b; Fumaric acid, C c,

for the temporary radio-activity exhibited by bodies left in the neighbourhood of thorium compounds. The successive changes—Thorium $\rightarrow$ Thorium X $\rightarrow$ Emanation $\rightarrow$ Matter† causing imparted or excited activity $\rightarrow$ The final product—go on simultaneously in thorium compounds, and the radio-activity ordinarily observed represents the combined effect of these different types of matter. According to Rutherford ("Radio-activity," p. 307), each of the components supplies approximately an equal proportion of the total activity. Thorium X and its disintegration products, however, constitute but an infinitesimal amount of the total quantity of matter, but their rate of transformation is immense in comparison with that of thorium.

* From the *Journal of Physical Chemistry*, ix., No. 8, p. 682.
† This type has been shown to be complex. Cf. Rutherford, *Phys. Zeit.*, 1902, 254; *Phil. Mag.*, 1903, [6], v., 95; von Lerch, *Drude's Ann.*, 1903, xii., 745; Pegram, *Phys. Rev.*, 1903, xvii., 424. This product is variously designated by different writers as the induced activity, the excited activity, the imparted activity, and a component of the secondary activity.

Rutherford and Soddy have succeeded by chemical means in removing from thorium compounds the matter causing the major part of the radio-activity. The thorium, however, still retains some activity, and within a month it recovers its normal equilibrium value. During the same time the separated non-thorium types of matter rapidly lose their radio-activity, and at the end of a month are comparatively inactive. (Compare curves A, a; Plate I.)

The separation of ThX from thorium consists in precipitating thorium as hydroxide from an aqueous solution of the nitrate by means of dilute ammonia, added in excess. The filtrate, free from thorium, after evaporation and ignition to remove ammonium salts, yields a minute residue of intense activity compared with that of the original thorium, weight for weight. The precipitate, although containing all the thorium, possesses no emanating power at first, and its activity is less than half of its original value. The matter causing the imparted activity, being insoluble in dilute ammonia solutions, is found in the precipitate. When thorium is precipitated as carbonate, oxalate, or phosphate, the residues obtained from the filtrates are inactive and the precipitates show no loss of radio-activity. In fact, ammonium hydroxide was the only reagent of those tried by Rutherford and Soddy which

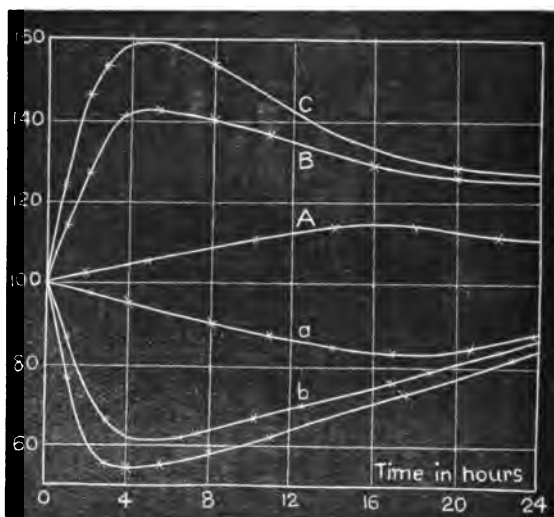


PLATE II.

separated ThX from thorium compounds, thus showing that ThX is insoluble in aqueous solutions of the reagents ordinarily used for precipitating thorium. With the view of arriving at other methods of separation we jointly undertook some experiments with thorium compounds, and, although work is still in progress, a report on some of the methods by means of which a separation is obtained may be of interest. In connection with this work we found a method of separating, by chemical means, the matter causing imparted activity from the other components, and these experiments are included in this paper.

Separation by means of Pyridine.

In the course of the preliminary experiments it was found that the separation of ThX from thorium is readily carried out by means of pyridine. When pyridine is added in excess to a dilute aqueous solution of thorium nitrate, no precipitate forms at room temperatures, but upon heating the solution, the thorium is completely precipitated as hydroxide (Miss Jefferson, *Journ. Am. Chem. Soc.*, 1902, xxiv., 545). The filtrate, after evaporation and ignition in order to expel the pyridine nitrate, yields a very slight residue which was found to be highly radio-

active. Its rate of decay is the same as that of ThX, its activity falling to half value in approximately four days.

Two samples of pyridine from Merck were used in these experiments. One was marked "free from homologues," and the other was the medicinal grade. The samples were re-distilled, the former passing over between $114.5-115.5^{\circ}$, the latter between $114-118^{\circ}$ under a pressure of 748 mm. The two samples exhibited no specific difference in their action with the thorium solutions. Three twenty-grm. samples of thorium nitrate, obtained from Eimer and Amend, New York, were used in the course of the experiments. A composite of each sample was prepared by pulverising it rapidly in an agate mortar. Subsequent hydration was guarded against, so that the small samples used in the separations should contain the same percentage of thorium. The nitrate upon ignition lost 51.3 per cent. in weight. Assuming that the sample was pure thorium nitrate, the calculated amount of water of hydration present was approximately 3.5 molecules per molecule of nitrate.

Some experiments were carried out for the purpose of ascertaining the conditions favourable for the separation of ThX by means of pyridine. Within a wide range the dilution of the thorium nitrate solution has little or no effect on the separation. Solutions varying in strength from 0.1 to 10 per cent, and containing the same amounts of thorium, yielded filtrates free from thorium, which after evaporation and ignition possessed approximately the same activity. For complete precipitation of the thorium the pyridine must be added in excess. The addition of pure pyridine gave the same results as an aqueous solution of the reagent. Boiling coagulates the gelatinous precipitate and causes it to settle rapidly. In one case the precipitate was boiled for half an hour, with an excess of pyridine in a flask fitted with a reflux condenser. The residue from the filtrate, however, did not differ materially in activity from the residue of another sample precipitated at the same time from a hot solution without boiling. Not all of the ThX in the sample is removed by one precipitation with pyridine. In removing ThX by means of ammonia, Rutherford and Soddy found that three successive precipitations were necessary to free thorium completely from ThX. The same holds true for the removal of ThX by means of pyridine. By dissolving the precipitate in hot dilute nitric acid and then re-precipitating the thorium with pyridine, as rapidly as possible, so that the ThX produced in the precipitate shall not become appreciable, residues were obtained from the successive filtrates with activities 100, 7.7, and 1.8 respectively. In three successive precipitations with ammonia, Rutherford and Soddy obtained residues whose activities were in the ratio of 100, 8, and 1.6 respectively, and in one of our experiments in which three successive precipitations with ammonia were made, the activities of the residues obtained were 100, 9.1, and 1.0 respectively. In several instances, however, the activities of the residues obtained from the second and third filtrates were considerably greater with both pyridine and ammonia than the values cited, although the conditions under which the precipitations were made were apparently the same.

The amounts of thorium nitrate used in the different experiments ranged from 0.2 to 1.0 gm., but as a rule a known weight of the nitrate was taken. With amounts greater than 0.2 gm. a suitable fraction of the filtrate was evaporated, platinum dishes being used for the most part. The precipitate was dried, together with the filter paper, on a hot iron plate. The greater portion was then removed from the paper and the latter incinerated. Ashless filter paper was always used. The entire precipitate was then transferred to a mortar and ground up under 95 per cent alcohol.* The contents of the mortar were next poured out upon shallow, circular, metal dishes, 12 c.m. in diameter. Particles of the precipitate adhering

to the mortar were washed out with alcohol, and the latter was removed by setting fire to it. A thin layer of thorium oxide, of nearly uniform thickness, was obtained in this way, which adhered sufficiently to the dish to prevent shifting of the powder in transferring it to and from the testing apparatus for the subsequent measurements of radio-activity. The amount of thorium oxide spread out on one of the dishes was approximately 0.1 gm.

The radio-activity of the precipitates and residues was determined by the electrical method, the Dolezalek type of quadrant electrometer being employed. When the needle was charged to 100 volts, a difference of potential of one volt between the two pairs of quadrants produced a deflection of 280 divisions on a millimetre scale, one metre from the mirror. The directions given by Rutherford in his book on "Radio-activity," for the measurement of ionisation currents by means of the quadrant electrometer, were found very helpful.

The testing apparatus consisted of a cylindrical vessel of galvanized iron, 15 c.m. in diameter and 30 c.m. high, supplied with an insulated central brass rod, to the lower end of which a circular disc 8 c.m. in diameter was soldered. The vulcanite insulator, carrying the central electrode, was provided with a guard ring to prevent leak across the insulation. The guard ring was kept earthed during the tests and was insulated from the testing vessel by means of sulphur and sealing-wax. The lower open end of the testing vessel fitted into a circular groove in a wooden base covered with thin sheet copper, and supported by vulcanite legs. The groove contained about 0.5 c.m. of mercury, which served as a seal. A potential of 150 volts applied to the testing vessel was found sufficient for obtaining saturation values for the ionisation currents measured. The electrometer, testing vessel, and connections were screened from external electrical disturbances by placing them in a cage of wire netting which was well earthed.

The connections were made in the usual way. One pole of a water battery of 156 cells was earthed, the other was connected to the outside of the testing vessel. From the central electrode of the latter a wire connected with one pair of quadrants of the electrometer, the other being earthed. The central electrode of the testing vessel also had an earth connection which was broken, when readings were made, by means of a special switching device (*Journ. Phys. Chem.*, 1905, ix., 324).

In measuring the radio-activity of the precipitates and residues, readings were made at short intervals—usually about an hour—during the first twelve hours after separation. Subsequently, measurements were made at intervals of gradually increasing length for a period of a month, at the end of which time the activity of the precipitate had acquired a constant maximum value, while the residue from the filtrate had become completely inactive. Between tests the dishes containing the samples were covered loosely with watch-glasses, and care was taken to disturb them as little as possible during the progress of their decay and recovery. On account of the emanation, equilibrium was often not established in the testing vessel for several minutes after the sample was introduced. The plan followed was to make readings until constant values were obtained. The effect due to the emanation was then determined by covering the sample with two thicknesses of ordinary paper, which cuts off nearly all the α -radiation (Rutherford and Soddy, *Journ. Chem. Soc.*, 1902, lxxxi., 837). The difference between the values calculated from the two readings was taken as the activity of the sample. The air-leak of the apparatus, about 25 m.m. divisions per minute, was deducted from the values found. As the measurements for any pair of samples usually extended over a month, and as slight changes in the sensitivity of the apparatus occurred, it was found necessary to employ the usual standard of uranium oxide in order to ascertain the corrections to be made in connection with the readings due to such changes.

In carrying out two parallel separations with ammonia

* The suggestion for this method was obtained from the work of McCoy on uranium (*Journ. Am. Chem. Soc.*, 1905, xxvii., 4).

and pyridine by three successive precipitations with each reagent and using the same weight of thorium nitrate, it was invariably found that the filtrate from the sample precipitated with pyridine gave a residue of greater activity than the filtrate residue from the sample precipitated with ammonia, while the activity of the precipitate was correspondingly less than that of the precipitate obtained with ammonia. These differences in activity were found to be due to the matter causing imparted activity. When thorium is precipitated with ammonia, Rutherford and Soddy found that the matter causing imparted activity is insoluble in dilute ammonia, and hence is found in the precipitate. When the separation is made by means of pyridine the matter causing imparted activity remains in solution—in part, at least, as the following experiment shows:—ThX was removed from thorium by three successive precipitations of a dilute aqueous solution of thorium nitrate with ammonia. The residue obtained by evaporating that portion of the filtrate given by the third precipitation, was tested for radio-activity and found to contain only about 1 per cent of the activity of the residue obtained by evaporating the filtrate from the first precipitation, thus showing that the ThX had been practically all removed. The thorium hydroxide precipitate, free from ThX, was at once re-dissolved in hot dilute nitric acid, rapidly re-precipitated with pyridine, and the precipitate filtered off by means of a suction-pump. The filtrate, after evaporation and ignition, gave a very minute residue, which was found to be radio-active. It was identified as the matter causing imparted activity by its rate of decay, its activity falling to half value in eleven hours.

Four successive precipitations then—three with ammonia and one with pyridine—separate ThX and a portion of the matter causing imparted activity.

(To be continued).

PROCEEDINGS OF SOCIETIES.

FARADAY SOCIETY.

Ordinary Meeting, December 12th, 1905.

Mr. JAMES SWINBURNE, Vice-President, in the Chair.

MR. J. SWINBURNE and Dr. G. RUDORF presented a paper on "*The Physics of Ore Flotation.*" (See CHEMICAL NEWS, vol. xcii., p. 288).

Prof. A. K. HUNTINGTON then read a paper on the same subject entitled "*The Concentration of Metalliferous Sulphides by Flotation.*" The paper also embodies the author's contribution to the discussion on the previous paper.

The paper has reference to the flotation processes of Potter and Delprat, which have been successfully applied in practice to the further concentration of the "tailings" obtained in the treatment of the sulphide ores of the Broken Hill district. These ores consist of sulphides of lead and zinc carrying silver, with a gangue composed of quartz, rhodonite, garnets, and small quantities of carbonates.

The flotation process consists in adding to the tailings dilute sulphuric acid (Potter), or a solution of acid sulphate of soda (Delprat) at about 65° C. Under these conditions the metallic sulphides are floated up by attached bubbles of gas, and form a coherent scum which can be removed, leaving behind the gangue, the separation being practically a quantitative one.

Experiments are described which prove that the gas causing flotation is CO₂ derived from native carbonates of iron and manganese present in the ore, and not from calcite or from carbonates produced on the surface of the sulphides by weathering. Carbonates which are decomposed by dilute sulphuric acid in the cold do not give rise to the formation of a scum.

Experiments are also described showing that the gas escaping during flotation carries an electrical charge, leaving an opposite charge on the solution. Electrometer determinations of the charge were made, but it was not found possible to establish a direct connection between the electrical phenomena and the fact of flotation. No change in the surface tension conditions of the solution in contact with a metallic sulphide is to be observed on electrification.

The assumption of Messrs. Swinburne and Rudorf of the presence of an air-film on the surface of the sulphide particles is criticised, and it is shown that the particles are floated perfectly after precautions have been taken to remove any adherent film of gas by exhaustion with acid, washing with alcohol, treatment with air-free distilled water, and exhaustion with the pump, &c. Flotation depending on the presence of an adhering film of air is made use of in certain suggested concentration processes, which, however, are different in principle from those under consideration.

It is a curious fact that specimens of zinc blende containing but little gangue, which do not form a scum on treatment with sulphuric acid and carbonate of iron, give an excellent flotation if previously mixed with sand. On the other hand, too great dilution with inert matter injures the flotation. The Broken Hill vanner tailings appear to be ideally suitable for the flotation process. These experiments dispose of Messrs. Swinburne and Rudorf's contention that blende scarcely floats at all, and incidentally of the statement that "zinc blende is thus easily raised by galena when intimately associated." They themselves admit that galena has to be very finely pulverised to float successfully; it is difficult therefore to see how it could be expected to float with attached inert blende. It appears more probable that the blende helps the galena to float. This view was strengthened by some experiments made on the wetting of sulphide surfaces.

The degree of wetting of a surface may be judged by measuring the angle which the liquid makes with it. Direct determinations of this angle for different minerals by a goniometer method are described, the values obtained varying from 47° to 63° for different sulphides. Quartz and magnetite are completely wetted by water. Alcohol wets all sulphides, but on again placing in water the apparent "greasiness" is restored.

When a gas bubble comes into contact with a mineral particle, the extent of surface of the bubble which will be in contact with the solid depends on the angle of contact of the solid with the liquid. If the angle is such as to cause a relatively large surface of the gas to be in contact with the solid, and the bubble is sufficiently large, the particle will be floated. The less the wetting the greater will be the force required to detach the bubble. If the surface of the solid is wetted, the bubble has no attachment at all. The different conditions met with are illustrated by means of diagrams.

Mr. BERTRAM BLOUNT said, although unable to put forward a satisfactory theory of his own, he was unconvinced by those proposed by the authors. Nor did their theories help in practice, for the only way to discover whether the flotation process could be applied to a particular ore was by experiment.

Mr. J. G. A. RHODIN thought that the crux of the theoretical difficulty was to discover how the gas bells were able to break through the liquid surface-films on the solid particles so as to come into contact with the latter; once that was known, Quincke's formula for triple contact of gas, liquid, and solid, could be applied. The key to the problem lay in the application of the law of mass-action, the necessary condition being that the solid must partly go into solution. The occluded or condensed gases which it is practically impossible to remove from the surface of a solid helped the action.

Dr. O. J. STEINHART referred to oil separation processes. He also pointed out that no separation process could be considered entirely satisfactory which did not separate galena from blende.

Mr. S. F. SPIERS described some measurements of the Volta effect between metals in so-called *vacuo*, which illustrated the difficulty, if not impossibility, of removing condensed air-films from the surfaces of solids.

Dr. F. MOLLWO PERKIN remarked on the differences between occlusion and surface-condensation of gases.

Mr. N. J. BLUMAN said that the theories put forward did not explain the selective action of the air-bubbles.

Mr. R. FREDERICK YORKE thought that an electrostatic theory would be required to explain the selective action.

Dr. H. BORNES hoped that more experiments would be made so as to explain some of the anomalous effects observed by Professor Huntington.

Dr. G. RUDORF said that Mr. Swinburne and he did not claim "weathering" as absolutely essential. With reference to the contact angle made by a liquid with a solid, that itself depended on the air-film.

Mr. J. SWINBURNE said that the essential point which the theory Dr. Rudolf and he had put forward was the temperature effect. No theory could explain the differences in the action on the different constituents of the ore any more than one could the differences of specific gravity; these depended on the varying inherent properties of matter. The presence of an air-film was not necessary, if the particles to be lifted were "greasy."

Prof. J. WALKER, F.R.S., contributed a paper on "*The Ions of Pure Water*."

In the discussion on Dr. Lowry's paper, on "*An Application to Electrolytes of the Hydrate Theory of Solution*," Mr. Bousfield drew attention to an apparent discrepancy between the temperature coefficient of the mobility of hydrogen and hydroxide ions on the one hand, and the temperature coefficient of the conductivity of water on the other. The author points out that water for which the data used by Mr. Bousfield holds good owes its conductivity almost wholly to impurity, and that Mr. Bousfield in his theoretical deduction has neglected the effect of temperature on ionic concentration. When the data obtained by Kohlrausch for pure water are employed, and when allowance is made for the temperature coefficient of ionisation, the discrepancy vanishes.

Mr. W. R. BOUSFIELD (communicated) said that he did not profess to be dealing with *pure* water, but with "water" in inverted commas. But he agreed now that there was strong evidence that pure water had a genuine conductivity due to H and HO ions.

NOTICES OF BOOKS.

Radio-activity. By E. RUTHERFORD, D.Sc., F.R.S., F.R.S.C. Second Edition. Cambridge: The University Press. 1905.

THE text of the most recent edition of this work has been extensively enlarged and re-arranged, the rapidity with which our knowledge of the subject is widening rendering it necessary almost to re-write the whole. At the same time, considering the imperfect state of development of the subject, it is especially noticeable how very seldom the author's predictions in the first edition have had to be corrected in accordance with increased knowledge, and how remarkably some of the theories he put forward have been verified. As a case in point, we may mention the disintegration theory, which has proved to be of the greatest value in explaining and interpreting the relations between the properties of the various radio-active substances. The new matter which has been added to the book relates specially to the theory of successive changes, which is treated fully in three fresh chapters, in which the mathematical theory is discussed very lucidly, and the results obtained by mathematical analysis are applied to explain the radio-active phenomena in the case of the elements uranium, radium, thorium, and actinium, and their products.

A large amount of new material dealing with the properties of the radiations and emanations has also been given a place in the book, and, in fact, besides the new chapters mentioned above, the last three chapters have been almost entirely re-written, while considerable alterations have also been made in the earlier parts. The book contains an admirable exposition of the subject, and will be found quite invaluable by those who are interested in the new science; they will find it full of the very latest information, while it need hardly be said that all theories are critically discussed by the author in the most unbiassed spirit.

The Laboratory Book of Dairy Analysis. By H. DROOP RICHMOND, F.I.C. London: Charles Griffin and Co., Ltd. 1905.

THIS book contains very explicit and practical directions for the analysis of milk, butter, and cheese, and although we should not recommend the untrained novice to attempt to embark upon technical analysis, he would undoubtedly find this work a very safe guide, and with its aid he might be able to ascertain a good deal of valuable information about the quality and condition of the above dairy products. This, however, applies only to the simpler tests, the more difficult being described in technical language for the benefit of the experienced chemist only. In some cases the experimental directions for these more difficult tests might with advantage have been made somewhat fuller; for instance, it seems doubtful whether the instructions relating to the estimation of sugar by the polarimetric method would enable anyone who was not already fairly conversant with its working to perform analyses. Speaking generally, however, the author has given careful attention to the details of manipulation, and the pages on the application of analytical methods to the solution of problems give a useful though slight account of the various problems with which the dairy chemist may be confronted. The treatment of butter and cheese is rather less full than that of milk, but instructions are given for the performance of complete ordinary analyses of both substances.

Terrestrial Magnetism and its Causes. By F. A. BLACK. London: Gall and Inglis. 1905.

THIS book puts forward a new theory to explain the fact that the earth is a magnet. Adopting the hypothesis that the space surrounding the earth, beyond its atmosphere, is permeated by a medium of an electrical nature, the author suggests that waves of this medium are impacted against the earth, each portion of which in consequence of its diurnal movement passes under these electric waves, and thus a "sheet of electrical influence" surrounds the earth, which is therefore magnetised by electric induction. Here, of course, a difficulty at once suggests itself, viz., that the magnetic poles should by this theory be identical with the orbital poles, which is not the case, and the author, fully aware of this objection, devotes a large portion of his book to proving that the magnetic poles describe a diurnal revolution resembling that of the orbital poles. It seems to us, however, that the author in this connection is too ready to adopt probabilities and likelihoods in place of established facts, and although he has collected and sifted a large amount of material bearing upon the magnetisation of the earth, we cannot see that he has really forwarded the elucidation of the question. The book is obviously the result of careful work undertaken from a genuine love of the subject, and as such deserves the attention of those who are devoting themselves to endeavouring to solve this complicated problem.

Zur Kenntniss der Kupfer-Zinklegierungen. ("A Contribution to Our Knowledge of the Copper-zinc Alloys"). By Dr. OTTO SACKUR. Berlin: Julius Springer. 1905.

THESE papers, which are reprinted from the *Arbeiten aus dem Kaiserlichen Gesundheitsamte*, contain detailed ac-

counts of the work which the author, assisted by Dr. P. Manz and Dr. A. Siemens, has done in reference to the question of the behaviour of metallic alloys towards acid liquids. The thorough investigation of this subject, which is one of great importance both from the scientific and hygienic standpoints, has brought to light some interesting new facts concerning the constitutions, solution pressures, and other physical properties of the alloys of lead with tin, and copper with zinc. The work on the former alloys has already been published, and is therefore given only in outline in this volume, while the copper-zinc alloys are treated more fully; from the investigation of these latter the author establishes the conclusion that zinc and its alloys with copper are passive in many solutions.

Nouvelles Méthodes Générales d'Hydrogénation et de Dédoublément Moléculaire. ("New General Methods of Hydrogenation and of Molecular Condensation"). By PAUL SABATIER and J. B. SENDERENS. Paris: Gauthier-Villars. 1905.

THIS small volume contains a detailed exposition of the authors' investigation of general methods of hydrogenation by means of reduced metals, describing the apparatus and experimental procedure, and discussing the principal results obtained. The metals employed include nickel, cobalt, iron, and copper, and by means of them many reactions have been induced; nickel and copper reduced from their oxides being specially active in this respect. These reactions, moreover, generally occur without any alteration in the appearance of the metal, which acts as a catalyser. In some cases the hydrogenation constitutes a complex chemical phenomenon; for instance, the hydrogen replaces oxygen, eliminated as water, or the halogens, eliminated as hydro-acids. The most important new method among the many transformations thus affected which the authors discuss, is perhaps the action of gaseous hydrogen in presence of reduced nickel upon volatile substances at temperatures above 250°. Numerous condensations are similarly produced, and the authors have arrived at somewhat unexpected results in the case of acetylene, from which they have obtained liquids closely resembling natural petroleum.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

Comptes Rendus Hebdomadaires des Séances de l'Académie des Sciences. Vol. cxli., No. 23, December 4, 1905.

Action of Silicon on Pure Aluminium, on Impure Aluminium. Silico-aluminides.—Em. Vigouroux.—The author investigates the condition of the silicon found in alloys of silicated aluminium, and particularly the nature of the metallic cement in which silicon crystallises when aluminium is allowed to act in excess on silicated substances, i.e., silica, fluorides, &c. Does the aluminium remain as such, or is it converted into silicide? He maintains that the silicide is not formed, for, on treatment with hydrochloric acid, aluminium chloride, hydrogen, and crystallised silicon are obtained instead of amorphous silicon. In the gas liberated from this reaction there is no siliciuretted hydrogen, and the presence of silica in small quantities in the liquid will be explained later. And, finally, the brown particles found in the solid residue, although apparently amorphous, can be shown by the microscope to be crystallised silicon. In the case of impure alloys of aluminium the results are quite different. Combustion can take place, due to the presence of a third element in the impurity, with the formation of compounds which the author calls "silico-aluminides." He was led to the discovery of these new bodies while trying to form crystalline silicides without the use of free silicon. By heating the double fluoride of potassium and silicon with aluminium in known proportions with a small quantity of the oxide of the alloying metal, cooling, and then

alternately treating the residue with hydrochloric acid and soda to destroy the free aluminium and silicon, the author obtained a silico-aluminide. In this way he has obtained silico-aluminides of iron, nickel, cobalt, chromium, manganese, molybdenum, tungsten, vanadium, uranium, titanium, &c., but has not succeeded, up to the present, in getting those of lead, tin, antimony, and bismuth. He then proceeded to determine three different methods of preparation, of which he gives details. As to the general properties of the silico-aluminides, they are comparable to the silicides; they have a metallic lustre, they are heavy, hard, brittle; their crystalline forms are often very definite. A certain number can be melted in the reverberatory furnace in a current of hydrogen. Acids attack a certain number, with the formation of silica, but most of those studied are not acted upon by acids, however concentrated, except by hydrofluoric acid. All resist the action of alkalis in solution. The author's conclusions are as follows:—
1. Silicon and aluminium, unable to combine in the purest state to form silicides of aluminium, often combine in the presence of a third metal, forming silicides of aluminium and that metal; in other words, silico-aluminides, definite crystalline bodies. 2. The knowledge of the cases in which these compounds are formed leads one to the rejection of any clay derivative as a recipient in all cases of preparation of metals susceptible to the formation of silico-aluminides, and explains why metals like vanadium, uranium, titanium, &c., have not been separated in a state of purity.

On Decahydronaphthol-a and Octohydride of Naphthaline A.—Henri Leroux.—1. *Decahydro-naphthol-a*, $C_{10}H_{12}-OH$.—When naphthol-a is hydrogenated below 200°, and the liquid mixture passed several times through a nickel tube, crystals are obtained which, after repeated crystallisation from petrol ether or acetone, give decahydronaphthol-a. This compound appears as fine colourless needles, melting at 62°, slightly soluble in water, but readily in the usual solvents. It distils at 230° at ordinary pressure without decomposing, and volatilises at 100° under 12 m.m. pressure. Heated in the presence of phosphoric anhydride, anhydrous oxalic acid, or fused bisulphate of potassium it loses one molecule of water, forming octohydride of naphthaline. *Octohydride of Naphthaline A.*—This compound the author provisionally calls octohydride of naphthaline A, formed from the dehydration of the α -decahydronaphthol, reserving the letter B for that obtained from the β -compound. It is a liquid distilling between 195° and 200°, mixed with decahydronaphthol-a. By treating an ether solution of the mixture with sodium, the decahydronaphthol forms an insoluble sodium derivative, which is easily separated off. By distilling off the ether, the pure carbide is left behind. It is a colourless liquid boiling at 190–191° at ordinary pressure; its density is 0.931 at 0° and 0.914 at 17°. The index of refraction for the D line is 1.4993 at 17°, corresponding to a molecular refraction of 43.71. The calculated index is 43.52.

NOTES AND QUERIES.

*. Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Fruit Essences.—Can any reader supply information as to the best works on the subject of the compounding of essences for the manufacture of non-intoxicating beverages?—N. E.

MEETINGS FOR THE WEEK.

MONDAY, 8th.—Society of Chemical Industry, 8. "Cinchona Barks and their Cultivation," by D. Howard. "New Method for the Quantitative Estimation of Acetone," by S. J. M. Auld.

TUESDAY, 9th.—Royal Institution, 3. (Christmas Lectures, adapted to a Juvenile Auditory). "Astronomy," by Prof. Herbert Hall Turner, D.Sc., F.R.S.

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PHYSICS	A. GRIFFITHS, D.Sc.
MATHEMATICS	E. H. SMART, M.A.
BOTANY	A. B. RENDLE, M.A., D.Sc.
ZOOLOGY	H. W. UNTHANK, B.A., B.Sc.
GEOLOGY & MINERALOGY	G. F. HARRIS, F.G.S.
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Spartelite (see NATURE, March 31, 1904, page 523), 2/- per piece.
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Flexible Sandstone, 8/- to 20/- (See NATURE, June 23, 1904, p. 185).
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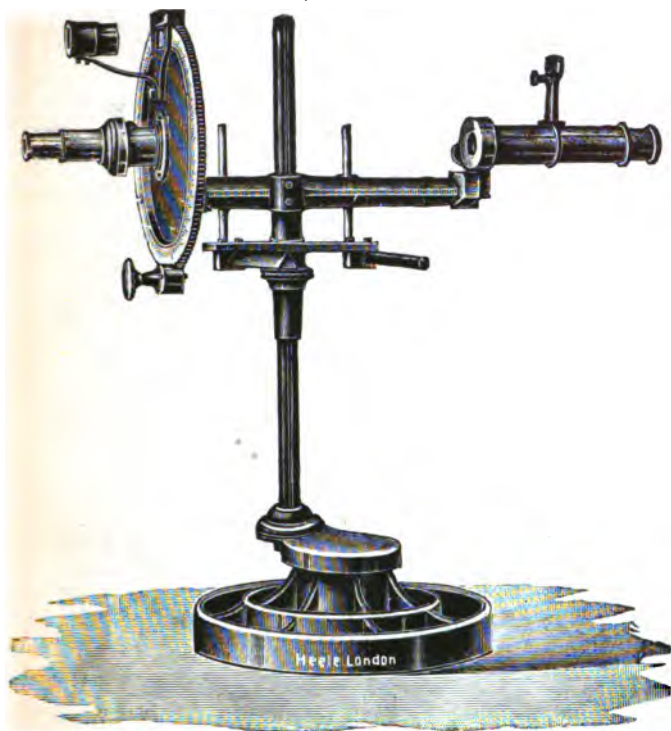
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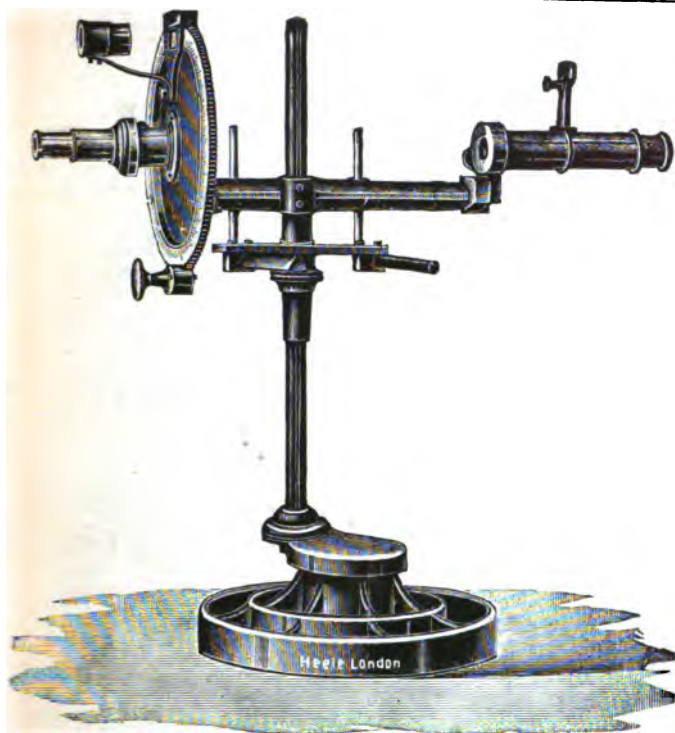
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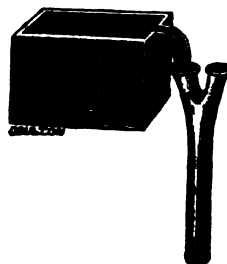
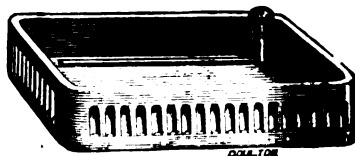
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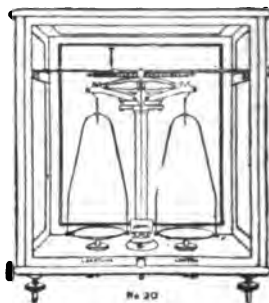
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NEW RESEARCHES ON THE ATOMIC WEIGHT OF NITROGEN.*

By PHILIPPE A GUYE.

(Continued from p. 5).

IV. *New Gravimetric Methods for Controlling the Atomic Weight of Nitrogen may take the place of the Classical Methods. They already admit of Determinations to a Unit of the Second Decimal place of the Atomic Weight of Nitrogen, and Confirm the Results of the Physico-chemical Values.*

HAVING proved that the chemical gravimetric methods are not capable of sufficient accuracy for determining the atomic weight of nitrogen to a unit in the second decimal place, and having also brought forward the remarkable agreement of physico-chemical methods applied to nitrogen, I considered that the conviction of chemists would only be satisfied with regard to this subject when the value $N = 14.01$ should have also been obtained by purely gravimetric methods.

The classical methods of Stas not yielding the desired degree of accuracy, it was essential to renew the study of this question by absolutely new methods, based upon the reliance of the atomic weight of nitrogen as directly as possible upon that of oxygen. In other words, it was necessary to revert to simple and rational methods, systematically discarding all indirect methods. The history of the determination of the atomic weights is rich in achievements of this kind; it will suffice to recall at this point the discovery by Dumas and Stas (1841) of an error of 2 per cent in the atomic weight of carbon, as fixed by Berzelius. The exact determination of two simple ratios— $C : CO_2$ and $CO : CO_2$ —by de Saussure's method was more conclusive than the numerous analyses of organic compounds previously carried out by the most famous chemists of their day.

Thus the problem reverts to the attempt to analyse accurately the oxygen compounds of nitrogen, which the best experimenters have generally regarded as impossible of exact execution. The difficulty of preparing these gases in the pure state is without doubt the chief reason that so far no attempt of the kind has been carried out. The researches undertaken in my laboratory since 1902 to attempt to fathom this abyss have proved that the difficulties are serious but not insurmountable, above all with the progress gained in technique relative to the manipulation of the gases. The results already obtained, it is true after many fruitless experiments, lead to absolutely conclusive numbers, which confirm the physico-chemical values.

Under these circumstances there is no doubt, either in the mind of my fellow-workers or in my own, that the improvements, which will finally be added to the new methods I am about to describe, will re-affirm the agreement of the physico-chemical and gravimetric values of the atomic weight of nitrogen.

In the first place, we embarked on the problem of the exact analysis of nitrous oxide, N_2O . This choice was chiefly influenced by three considerations:—

The first was inspired by the fact that the determinations of the exact density of this gas by different observers has given concordant results; this was a favourable presumption of the possibility of obtaining this body in a pure state.

The second was drawn from the high percentage of nitrogen in the sub-oxide, the highest among the oxygen

compounds of nitrogen; for the same accuracy, the atomic weight of nitrogen deduced from analysis of an oxide of nitrogen is more certain in proportion to the percentage of nitrogen contained in the oxide.

The third is based upon the fact, worthy of note, that the discussion of the causes of error in the experimental methods adopted, tends to prove that the value of the atomic weight of nitrogen thus obtained should be rather too great than too small. This point was of importance in view of the nature of the question under consideration.

The choice of nitrous oxide being settled, its analysis was effected in two ways—(a) gravimetrically, (b) volumetrically. The first experiments were made with the co-operation of Bogdan. Jaquerod and Bogdan also carried out the volumetric analysis.

Gravimetric Analysis of Nitrous Oxide.—This analysis was carried out by decomposing a known weight of the oxide by means of red-hot iron; the increase of the weight of the iron gave the weight of oxygen contained. It would take too long to enter into all the details of the experiment; I will confine myself to mentioning a few characteristic features.

In order to avoid the causes of error arising from the weighing of large bulbs, the nitrous oxide was weighed in the condensed condition in charcoal; one can thus work with small pieces of apparatus.

The decomposition of the oxide was carried out in an apparatus consisting essentially of a glass tube containing an iron spiral fixed at its extremities to two platinum bands passing through the tube; the tube is furnished with two small side-tubes fitted with perfectly air-tight taps.

Before the experiment the tube with the iron spiral was completely exhausted of air and weighed empty.

The analysis of the nitrous oxide consisted in causing the N_2O gas, set free from the tube with charcoal, to pass very slowly over the iron spiral, raised to incandescence (red-hot) by the electric current.

At the end of the experiment, having shut off the charcoal tube, a vacuum was made in the spiral tube by connecting the lower delivery tube to the mercury pump and continuing to heat the iron spiral, in order to prevent the fixation of nitrogen as nitride of iron.

All the weighings were carried out with counter-weights of the same glass and of practically the same volume.

For the details, I refer you to the complete memoir, only transcribing here our results.

TABLE XIII.—Atomic Weight of Nitrogen from the Gravimetric Analysis of Nitrous Oxide.

Nitrous oxide.	Oxygen.	Atomic weight N.
1.1670	0.4242	14.009
0.9498	0.3453	14.005
0.8652	0.3145	14.008
1.2247	0.4455	13.992
1.4202	0.5159	14.007
Mean		14.007

The mean weight of oxygen weighed being 0.4 grm., corresponding to 1.1 grm. of suboxide, the gravimetric errors are, if added, $\frac{1}{4000} + \frac{1}{11000} = \frac{3.4}{10000}$, and if they

compensate one another $\frac{1.6}{10000}$, giving a mean of $\frac{2.5}{10000}$.

The possible error ΔN resulting on the atomic weight of nitrogen will therefore be approximately:—

$$N = \pm \frac{44}{2} \cdot \frac{2.5}{10000} = \pm 0.0055.$$

The conduct of these experiments is very delicate. We have had many failures, also Bogdan and I have sought to give this method of analysis a more precise form. After several attempts, we believe we have found a more perfect method than the former. It consists in arranging an iron spiral, heated electrically, in a glass receiver furnished with a single ground-glass stopper; this apparatus is

* From the *Bulletin de la Société Chimique de Paris*, 1905.

weighed, first exhausted of air, then filled with nitrous oxide; the spiral is then raised to incandescence, allowed to cool, again exhausted, and weighed a third time.

With a trial apparatus of small dimensions containing only 0.4 grm. of nitrous oxide, the following figures were obtained:—

N.
14.00
14.03
13.98

Mean . . . 14.004

The possible error on the atomic weight of nitrogen resulting from the uncertainty of the weight of the oxygen (about 0.1450 grm.) and of the corresponding nitrous oxide is—

$$\Delta N = \pm 0.015.$$

Until we get nearer to the desired limit of accuracy (one unit in the second decimal place of the atomic weight of nitrogen), no conclusion can for the moment be deduced from this preliminary result.

These experiments should be resumed with apparatus of greater dimensions, the construction of which has so far presented very great difficulties.

(To be continued).

THE PHYSICAL AND CHEMICAL PROPERTIES OF IRON CARBONYL.*

By Sir JAMES DEWAR, M.A., Sc.D., LL.D., F.R.S.,
Jacksonian Professor in the University of Cambridge,

and
HUMPHREY OWEN JONES, M.A., D.Sc., Fellow of Clare College, and Jacksonian Demonstrator in the University of Cambridge.

(Continued from p. 3).

Vapour Density and Dissociation.—Two vapour density determinations were made by Mond and Langer (*loc. cit.*) by V. Meyer's method in an atmosphere of hydrogen at the temperature of boiling xylene. The results were 93.8 and 92.4, the theoretical value is 98, so that slight dissociation is indicated.

A number of trial experiments showed that iron carbonyl vapour dissociated quietly without explosion when heated to a high temperature alone or in an inert gas.

A series of vapour density determinations were made by V. Meyer's method in various gases at different temperatures in order to show the effect of temperature, of admixture with inert gas or carbon monoxide, and of the rate of diffusion on the dissociation of iron carbonyl. Owing, however, to the great stability of the iron compound and its slow rate of dissociation, these effects are not so clearly seen as in the case of nickel carbonyl. In fact, so slowly does iron carbonyl dissociate, that accurate determinations can only be obtained at the lowest temperatures where no dissociation occurs and at the highest temperatures where complete dissociation is rapid; at the intermediate temperatures the dissociation is so slow that diffusion to the cooler parts of the tube takes place and the end-point of the evolution of gas is extremely uncertain.

Several series of determination of the vapour density were also made by Hofmann's method to show the effect of pressure and temperature on the dissociation of iron carbonyl; but again, owing to the slow rate of dissociation, the experiments had to be prolonged for an inconveniently long time before even an approximately constant volume was obtained. In these experiments it is advisable to protect the vapour as far as possible from light, since the decomposition of the iron carbonyl by light, which takes place at low temperatures, is thus minimised.

The results obtained are tabulated below. The percentage dissociation is calculated by means of the formula—

$$p = \frac{D - d}{4d} \cdot 100.$$

Vapour Densities determined by Meyer's Method.

Temperature of the bulb.	Gas filling the tube.	Vapour density (H=1).	Percentage of Fe(CO) ₅ dissociated.
129° C. (amyl alcohol)	Carbon monoxide	93.7	1.2 (a)
		93.2	
	Nitrogen	90.1	2.2
	Hydrogen	86.2	
155° C. (turpentine)		88.7	3.0 (b)
		87.5	
	Carbon monoxide	82.6	4.6
	Nitrogen	78.0	6.4
182° C. (aniline)		36.4	42.3
	Carbon monoxide	44.9	30.1
		44.0	
	Nitrogen	27.4	64.4 (c)
216° C. (naphthalene)	Carbon monoxide	20.2	96.3
	Nitrogen	20.0	97.5

Vapour Densities by Hofmann's Method.

Temperature.	Pressure.	Vapour density (H=1)g.	Percentage of Fe(CO) dissociated.
78° C.	195	99.8	—
	195	98.4	
	212	100.0	
	288	99.8	
100° C.	126	98.3	—
	179	98.6	
	204	97.2	
	225	97.1	
	298	99.5	
130° C.	136	95.0	0.8
	192	95.7	0.6
	216	96.2	0.5
	242	94.5	0.9
	325	95.0	0.8
141° C.	261	86.6	3.3
155—160° C.	274	70.2	9.9
	354	88.1	2.81
179° C.	249	40.4	35.6 (c)
	334	40.4	
	406	44.2	
	574	45.6	

Remarks.

- No visible deposit of iron.
- Distinct deposit of iron.
- Extensive deposit extending over large area of tube.
- Extremely faint deposit of iron.
- Very extensive deposit of iron; on cooling much undecomposed iron carbonyl condensed.

These results show very clearly the effect of increase of temperature and of diminution of pressure in increasing the dissociation, the effect of carbon monoxide in diminishing the dissociation observed in an inert gas at the same temperature. The increased dissociation in a light inert gas as compared with a heavy one is shown by the values in hydrogen and nitrogen at 129° C.

The rate of dissociation of the vapour at various temperatures was also studied in an apparatus very similar to that used by Mittasch for the dissociation of nickel carbonyl (*Zeit. Phys. Chem.*, 1902, vol. xl., p. 1). Owing to the small vapour pressure of iron carbonyl the range of pressures of undissociated vapour is very limited.

The rate of dissociation is very slow at temperatures below 180° C., but appears to be complete at that temperature. The reaction is reversible, but the reversal takes place very slowly.

* A Paper read before the Royal Society, November 16, 1905.

Chemical Reactions of Iron Pentacarbonyl.—An examination of some of the simpler reactions of iron carbonyl was made in order to study its chemical nature and stability in comparison with nickel carbonyl, the reactions of which have already been described (*Trans. Chem. Soc.*, 1904, vol. lxxv, pp. 203–222). The action of the halogens in solution in pure dry carbon tetrachloride on normal and decinormal solutions of iron carbonyl in the same solvent was first investigated. The solutions were mixed in a nitrometer and allowed to stand, the gas evolved measured and tested, and the solid examined.

Chlorine and iron carbonyl react fairly rapidly to produce a solid and carbon monoxide, which was measured and showed that the decomposition was complete. The solid was found to be a mixture of ferrous and ferric chlorides. The ferrous chloride could never be obtained quite free from ferric, but by using a large excess of chlorine practically pure ferric chloride could be obtained. The gas appeared to be practically pure carbon monoxide and to contain no carbonyl chloride.

Bromine reacts with iron carbonyl very slowly—in fact, much more slowly than iodine reacts with nickel carbonyl. Five c.c. of a decinormal solution of iron carbonyl and 1 c.c. of normal bromine solution should evolve 22.4 c.c. of gas; 15 c.c. of gas had been evolved in six hours, 19.5 c.c. in 20 hours, and the reaction was practically complete in 35 hours. The solid salt consisted chiefly of ferrous bromide with minute traces of ferric salt; the gas was pure carbon monoxide. This reaction shows the much greater stability of the iron compound over the nickel compound, which is decomposed by bromine in a few seconds after mixing.

Iodine reacts extremely slowly with iron carbonyl to produce carbon monoxide and ferrous iodide; in decinormal solution the reaction had only proceeded to the extent of 70 per cent in three days.

Iodine monochloride in chloroform solution reacts with iron carbonyl much in the same way as it does with nickel carbonyl; ferrous chloride is precipitated and iodine liberated, the solution becoming purple; the free iodine then reacts very slowly with the residual iron carbonyl.

The whole reaction is a very slow one, and the salt at first precipitated is ferrous chloride, with no trace of ferric salt and no iodide; a little iodide appears before the formation of chloride is complete, but no ferric salt is produced.

Iodine trichloride in chloroform solution reacts slowly with iron carbonyl, producing a solid deposit and evolving gas; no free iodine is produced for a long time. The solid is again ferrous chloride free from ferric chloride, and contains very little iodide until the reaction has been proceeding for some time.

Cyanogen gas does not appear to react at all with iron carbonyl liquid or vapour, and in alcohol solution the reaction is extremely slow.

Cyanogen iodide in chloroform solution reacts very gradually, the solution first becoming red and then purple, and a brown solid is deposited. The solvent contains free iodine and the solid is ferrous cyanide mixed with a little ferrous iodide.

The action of the halogen hydrides was examined in the gaseous state and also in chloroform solutions.

Hydrochloric and hydrobromic acid gases had no action on iron carbonyl, even after allowing the mixture to stand for weeks in the dark. Dry **hydriodic acid** gas was introduced into an exhausted glass bulb containing a small glass bulb full of iron carbonyl which was then broken. On standing, a dark brown crystalline solid was produced, which was found to be ferrous iodide, and at the same time carbon monoxide and hydrogen were liberated.

In chloroform solution hydrochloric acid and hydrobromic acid react slowly with iron carbonyl, the former more slowly than the latter. In each case pure ferrous salts were produced and hydrogen and carbon monoxide were evolved. With a chloroform solution of hydriodic acid the reaction was more rapid and was complicated, as in the case of nickel carbonyl, by the liberation of iodine.

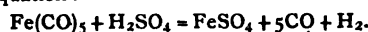
The reactions of iron carbonyl described above are exactly parallel with the corresponding reactions of nickel carbonyl, only differing in rapidity, which is much less owing to the greater stability of the iron compound. There is a more marked difference to be observed in some of the other reactions.

With **sulphur** dissolved to carbon bisulphide or xylene, iron carbonyl does not react at all in the cold, neither does it react with **nitric oxide**, in both cases differing markedly from nickel carbonyl.

Hydrogen sulphide also has no action on iron carbonyl, but an alcohol solution of the gas reacts extremely slowly to produce ferrous sulphide, carbon monoxide, and hydrogen.

Nitric acid in carbon tetrachloride or ether solution reacts rapidly with iron carbonyl to produce a mixture of ferrous and ferric nitrates, carbon monoxide with some hydrogen and reduction products of nitric acid.

Concentrated sulphuric acid reacts rapidly with iron carbonyl—the mixture first darkens, carbon monoxide and hydrogen are evolved, mixed with a little iron carbonyl vapour; the liquid then becomes paler in colour as the reaction is completed. The salt formed is pure ferrous sulphate, so that the reaction may be expressed by the following equation:—



The rapidity of the reaction is very much greater than in the case of nickel carbonyl; this is the only reaction yet observed which is more rapid than the corresponding reaction with nickel carbonyl.

To complete the comparison between the nickel and iron compounds in so far as the reactions of nickel carbonyl have been described, the reaction of iron carbonyl with **benzene** in presence of **aluminium chloride** was examined. Four grms. of iron carbonyl and 10 grms. benzene (five molecules) were poured on to 28 grms. powdered aluminium chloride (five molecules) in a tube, which was then sealed off and heated for two hours to 100° C. The mixture became very dark in colour, and on opening the tube some pressure caused by carbon monoxide and a little hydrochloric acid gas was observed.

The dark mass was mixed with ice, when some ferrous chloride dissolved and some ferrous hydroxide was precipitated, treated with hydrochloric acid and distilled in steam. Benzaldehyde and benzene came over first, followed by a crystalline fluorescent solid; the residue in the flask was dissolved in benzene, dried, and distilled, when a crystalline solid was obtained which was found to be pure anthracene, so also was the small quantity of solid which distilled in steam.

The final products of the reaction in this case are, therefore, precisely the same (namely, benzaldehyde and anthracene) as those produced by using nickel carbonyl. In the cold the reaction proceeds slowly, with the production of benzaldehyde and no anthracene, again precisely as in the case of nickel carbonyl.

(To be continued.)

Royal Institution.—On Tuesday next, January 16, at 5 o'clock, Professor E. H. Parker will deliver the first of a course of three lectures at the Royal Institution on "Impressions of Travel in China and the Far East"; on Thursday, January 18, at the same hour, Canon Beeching commences a course of two lectures on "Shakespeare"; on Saturday, January 20, at 3 o'clock, Mr. J. E. C. Bodley delivers the first of two lectures on "The Church in France." The Friday Evening Discourse on January 19 will be delivered by Professor J. J. Thomson, the subject being "Some Applications of the Theory of Electric Discharge to Spectroscopy"; on January 26 the discourse will be delivered by Mr. A. C. Benson, on "Walter Pater"; and on February 2 by Professor S. P. Thompson, on "The Electric Production of Nitrates from the Atmosphere."

THE CHEMICAL SEPARATION OF THE RADIO-ACTIVE TYPES OF MATTER IN THORIUM COMPOUNDS.*

By HERMAN SCHLUNDT and RICHARD B. MOORE.

(Continued from p. 9).

Separation by means of Fumaric Acid.

In the course of our qualitative experiments several of the methods recently worked out for the quantitative separation of thorium from some of the other rare earth metals were tried. In this connection we found the contributions from the quantitative laboratory of Columbia University on the separation of thorium from cerium, lanthanum, and didymium very suggestive and helpful (Metzger, *Journ. Am. Chem. Soc.*, 1902, xxiv., 901; Neish, *Ibid.*, 1904, xxvi., 780).† The separation of thorium from the other metals named, by means of fumaric acid, as worked out by Metzger, when carried out with thorium compounds alone, effects a separation of some of the radio-active components. This fact was first noticed by Baskerville, who did not, however, identify the products (*Journ. Am. Chem. Soc.*, 1904, xxvi., 931). Metzger found that thorium is precipitated quantitatively from a neutral 40 per cent alcoholic solution by means of fumaric acid, while cerium, lanthanum, and didymium remain in solution. When thorium was precipitated by means of fumaric acid from a solution of thorium nitrate, ThX was found in the filtrate, together with some of the matter causing imparted activity. The precipitates and residues obtained were treated in the same manner as the products of the pyridine separation. The ThX was completely removed by two successive precipitations. The second precipitation was carried out by re-dissolving the first precipitate in hot dilute nitric acid, evaporating to dryness at 150°, re-dissolving in hot 40 per cent alcohol, and re-precipitating with fumaric acid. The activity of the residue obtained from the second filtrate was extremely small when compared with that of the residue obtained from the first filtrate, and it was found that one precipitation was sufficient to obtain the results presented below, provided the precipitate was thoroughly washed with hot 40 per cent alcohol containing a little fumaric acid. The activity of the residue obtained by evaporating the filtrate and igniting, to decompose the excess of fumaric acid, was found to exceed that obtained from the same amount of thorium nitrate, when the separation was carried out by means of pyridine, while the activity of the precipitate was correspondingly less. This difference was found to be due to the presence in the filtrate from the fumaric acid precipitate of a greater amount of the matter causing imparted activity. That a portion of this matter which causes the imparted activity had been separated from the thorium was shown in the following manner:—A sample of thorium nitrate, after undergoing three successive precipitations without lapse of time, with ammonia, when precipitated a fourth time with fumaric acid and the filtrate evaporated and ignited, yielded a minute residue of considerable activity. The residue was much more active than that obtained when the fourth precipitation was made with pyridine instead of with fumaric acid. The same amount of thorium nitrate was used in the two cases, and the time required for the experiments was approximately the same. During the first few hours after precipitation the activity of the residue increased in value, reaching a maximum in about four hours, when it decreased according to an exponential law, falling to half value in eleven hours. The Curve D, Plate I., which shows the change of activity with the time, has the form characteristic for the radio-active type of matter deposited on a negatively-charged plate

when exposed for a short time—about fifteen minutes—to the emanation of thorium oxide. The type of matter causing imparted activity was also separated from thorium compounds by first precipitating the thorium from the solution by means of fumaric acid, filtering, and then adding dilute ammonium hydroxide in excess to the filtrate, to which a drop of lead nitrate or ferric chloride solution had been added previous to the addition of ammonia. The small precipitate obtained contained the matter causing imparted activity which decays to half value in eleven hours, after passing through a maximum at approximately four hours from the time of precipitation. (Curve D, Plate I.)

Curves of Recovery and Decay.

The changes in radio-activity of the types of matter separated from thorium compounds by the foregoing methods are represented graphically in Plates I. and II. Curves *a*, *b*, and *c*, Plate I., represent the recovery of activity of the precipitates with the time, when the separations are carried out respectively with ammonia, pyridine, and fumaric acid. Curves *A*, *B*, and *C* show the decay of activity of the radio-active products obtained by evaporating the filtrates corresponding to the respective precipitates. In Plate II. the initial portions of the curves of decay and recovery are plotted on a larger scale, but the letters refer to the same products as in Plate I. Curve D, Plate I., shows the decay of activity of the matter causing imparted activity, which was removed by the two methods described above in connection with the fumaric acid separation. The curves represent, in the case of ammonia, three precipitations, with pyridine two, and with fumaric acid one.

In plotting the results the plan of Rutherford and Soddy (*Journ. Chem. Soc.*, 1902, lxxxi., 839) was followed. The activities are represented on the axis of ordinates by an arbitrary scale. In Plate I. the final value of the precipitates* was taken as 100 in each case. The recovery curves *a*, *b*, and *c* were plotted on this basis. In plotting the curves *A*, *B*, *C*, showing the decay in activity of the residues, the initial value is represented by 100, time being reckoned from the time the precipitation was made. In the case of Curve D, Plate I., the maximum value of the activity was placed at 100, and the time scale represents hours instead of days. Curves *A*, *B*, *C*, *a*, *b*, *c*, Plate II., are plotted on the basis of the initial values of the activities, both the precipitates and residues being taken as 100.

Neglecting for the present the initial irregularities of the curves of decay and recovery in Plate I.—a full discussion of which is given further on—it appears from the close parallelism of the curves after the second day that the same radio-active process is taking place in each set of residues and precipitates. After the second day the three residues have approximately the same activity. The same is true of the precipitates. The activity of the residues decays according to an exponential law, falling to half value in about four days—a rate of decay characteristic of ThX. In each separation the initial activities of the precipitates are about the same, values varying from 41 to 46 having been obtained in different experiments. These precipitates recover their activity in a geometric progression with the time, attaining a maximum value within a month, which is due to the formation of ThX and its disintegration products. When the recovery curves are extended back they cut the axis of ordinates at about twenty, which represents the proportion of the final activity due to the “non-separable” activity of thorium, which as Rutherford and Soddy have pointed out, may be regarded as a distinct type of matter resulting from the slow disintegration of the inactive thorium itself, and very closely allied to thorium in its properties, or it may be regarded as the radio-activity due to thorium itself. The value of the non-separable activity found by us is somewhat less than that given by Rutherford and Soddy, viz., 25 per cent; but in this connection it must be noted that these investigators record a value of

* From the *Journal of Physical Chemistry*, ix., No. 8, p. 682.

† The contributions from the Harrison Laboratory, University of Pennsylvania, on the use of organic bases in precipitating and separating the rare earth metals likewise indicated other methods. (See *Journ. Am. Chem. Soc.*, 1902, xxiv., 540; 1903, xxv., 421, 1128).

* The effect due to the emanation is not included in the values.

22 per cent for the non-separable portion in one of their experiments. Inactive thorium was not obtained in the course of the separations. Hoffman and Zerban (*Ber.*, 1903, xxxvi., 3093) and Zerban (*Ibid.*, 1903, xxxvi., 3911; see also Baskerville and Zerban, *Journ. Am. Chem. Soc.*, 1904, xxvi., 1642), state that inactive thorium was obtained from the mineral gadolinite. On the basis of the non-activity of thorium the activity of thorium compounds must be ascribed to a radio-active type of matter generally associated with thorium and very like it in its behaviour towards different reagents. If this view should prove to be the correct one, then—as Rutherford and Soddy have pointed out—the rôle played by the non-separable activity in the theory of radio-active changes would simply have to be assigned to the new radio-active matter so closely resembling thorium in its other properties.

Coming now to the consideration of the initial irregularities in the curves of decay and recovery, it is seen that the residues obtained from the filtrates in the fumaric acid and pyridine separations increase in activity considerably more than the ammonia residue. Moreover, the fumaric acid and pyridine residues attain their maximum after five hours, while the ammonia residue only reaches a maximum after nearly twenty hours. On the other hand, the precipitates obtained with fumaric acid and pyridine drop in activity more rapidly at first than the ammonia precipitate, the curves passing through minimum values at the same time that the curves of decay of the corresponding residues attain maximum values. These differences in the curves are more evident from the plotting in Plate II., which shows simply the initial portions of the curves.

The maximum and minimum in the curves of decay and recovery have been satisfactorily explained by Rutherford and Soddy when the separation is carried out by means of ammonia. Their explanation is briefly as follows (Rutherford, "Radio-activity," pp. 295–299; Soddy, "Radio-activity," pp. 117–121):—Before any separation is made the activity observed is the sum of the several activities of (1) thorium, (2) ThX, (3) emanation, (4) matter causing imparted activity, the last named consisting of at least two stages recently designated by Miss Slater as ThA and ThB respectively (*Phil. Mag.* [6], ix., 628, May, 1905). The separation with ammonia yields a precipitate free from ThX and without emanating power at first. In the precipitate is found the thorium and the matter causing imparted activity (both stages). Immediately after the separation then neither the residue from the filtrate nor the precipitate is in radio-active equilibrium. Since the precipitate is free from ThX, which produces the matter causing imparted activity, via the emanation, but little imparted activity will be produced during the first day, because comparatively little ThX is present. In view of the very rapid change of the emanation into the matter causing imparted activity—one-half changing in about one minute—no serious error is introduced by omitting this step in an explanation of the initial irregularities of the curves. The imparted activity present in the precipitate, however, decays, and since its rate of decay is faster than the rate at which ThX is formed, the activity of the precipitate decreases for a time until the activity of the ThX and its disintegration products compensate the loss of activity due to the decay in the matter causing imparted activity. Rutherford and Soddy established the correctness of this view by separating ThX from thorium twenty-three times during a period of nine days, so as to remove the ThX nearly as fast as it was formed, while at the same time the matter causing imparted activity decayed. The recovery curve then obtained showed practically no drop, and finally after about a month attained a constant value approximately four times its initial value.

The initial rise in the ThX curve is explained by considerations similar to those advanced for the decay in activity of the precipitate during the first day. ThX changes into the matter causing imparted activity, and as the latter disintegrates more rapidly than ThX, the activity due to the imparted activity more than com-

pensates for the decay in activity of ThX itself; and the combined effect of these two activities gradually increases during the first day after precipitation, the maximum lying about 18 per cent higher than the initial value. The theoretical curve showing the combined effect of two radio-active types of matter, the first producing the second, and having respectively the radio-active constants of ThX and the imparted activity considered by them, is given by Rutherford on p. 299 of his book on "Radio-activity" (see also Curve A, Plate I.). The observed and theoretical values show a very good agreement.

(To be continued).

THE VOLUMETRIC ESTIMATION OF CYANATES.*

By A. C. CUMMING, B.Sc., and ORME MASSON, F.R.S.

(Concluded from p. 6).

As it was impossible to obtain perfectly pure cyanide or cyanate from which to make up mixtures of known composition, we decided to test the method by applying it first to separate stock solutions (a) of cyanide containing traces of carbonate and cyanate, and (b) of cyanate containing a trace of cyanide and a fair amount of carbonate, and then to apply it to mixtures of these in different proportions. If the method is trustworthy, the results obtained with the mixtures should agree with those calculated from the first analyses of the stock solutions; but there should be no such agreement if any or all of the analyses are incorrect. It seemed useless to attempt to check the results by independent analytical methods, as we know of none that can be regarded as wholly satisfactory.

In the first place stock solutions were made of approximately 0.1 N. KCN and 0.066 N. KCNO. Two mixtures of these were made, one (A) containing equal volumes, the second (B) containing 4 volumes of the former to 1 of the latter. The results were as follows:—

		Cyanide Solution.				
		I.	II.	III.	Mean.	
CN ..	..	0.0986	0.0984		0.0985	
½CO ₃ ..	..	0.0020	0.0017		0.0019	
CNO ..	..	0.0018	0.0003		0.0010	
		Cyanate Solution.				
CN ..	..	0.0019	0.0019	—	0.0019	
½CO ₃ ..	..	0.0256	0.0246	0.0246	0.0249	
CNO ..	..	0.0648	0.0663	0.0663	0.0658	
		Mixture A.			Mean.	
		I.	II.	III.	Found.	Calculated.
CN ..	0.0499	0.0499	—	0.0499	0.0502	
½CO ₃ ..	0.0141	0.0146	0.0139	0.0142	0.0134	
CNO ..	0.0329	0.0323	0.0334	0.0329	0.0334	
		Mixture B.				
CN ..	0.0777	0.0779	—	0.0778	0.0792	
½CO ₃ ..	0.0065	0.0068	0.0063	0.0065	0.0065	
CNO ..	0.0161	0.0143	0.0142	0.0149	0.0140	

Free alkali present, if any, was included in the figures given for carbonate. The cyanide was titrated with silver nitrate in the usual way, and the total alkali and the cyanate were determined by titration with congo indicator, as described in the previous part of this paper.

To ascertain whether the method would work in the case of stronger solutions, some of about ten times the former strength were made up and tested before and after mixing. The results were as follows:—

* Communicated by the Authors. From the *Proceedings of the Society of Chemical Industry of Victoria*, July–August, 1903.

Cyanide Solution.

	I.	II.	III.	Mean.
CN	0.978	0.978	—	0.978
$\frac{1}{2}$ CO ₃	0.026	0.032	0.032	0.030
CNO	0.013	0.015	—	0.014

Cyanate Solution.

	I.	II.	III.	IV.	V.	VI.	Mean.
CN	0.005	0.005	—	—	—	—	0.005
$\frac{1}{2}$ CO ₃	0.083	0.082	0.082	—	—	—	0.082
CNO	0.630	0.619	0.632	0.652	0.638	0.634	0.634

Mixture (Ten vols. Cyanide to One vol. Cyanate).

			Mean.	
	I.	II.	Found.	Calculated.
CN	0.890	0.890	0.890	0.889
$\frac{1}{2}$ CO ₃	0.037	0.037	0.037	0.035
CNO	0.073	0.073	0.073	0.070

These results seem to justify the recommendation of the method for the analysis of commercial samples of potassium or sodium cyanide. Certain precautions are, however, necessary.

In the first place, grave errors may be introduced in the calculation of the carbonate from the total alkali by subtraction of the cyanide, unless the acid used is reduced to the same true standard as the silver nitrate. Any small difference that may exist is thrown upon the carbonate. We prefer to use HCl standardised with Iceland spar and AgNO₃ standardised with the CaCl₂ solution obtained in the same operation (Masson, CHEMICAL NEWS, lxxxi., 73).

In the second place, the presence of much cyanate undoubtedly introduces some difficulty in the judging of the end-point in the cold titration with acid for total alkali. The best results are obtained by considerably diluting the solution, and running in the standard acid very slowly and with frequent agitation. This prevents local decomposition of cyanate by excess of acid before the true end-point is reached. Another obvious advantage of large dilution is that the evolution of HCN is less troublesome. We have made a few tests to compare the results obtained for carbonate as above with those got by the method first recommended (we believe) by Mellor, viz., the titration with acid of the faintly-clouded solution of KAg(CN)₂ obtained in the cyanide titration. This method, using congo as indicator, gives a very sharp end-point. The results seem to be slightly lower than those got by direct alkalimetry of the KCN solution, but we cannot say definitely that they are more accurate.

Thirdly, impure commercial cyanides may of course contain various substances which would tend to spoil the carbonate and cyanate determinations. As an instance, we find that much ferrocyanide is fatal, though traces do not seem to have any notable influence. But at the present day commercial cyanide is, as a rule, of high quality.

The cyanate values in the preceding tables were all obtained by the back titration after boiling with excess of acid, and not by the loss of NH₃, which results later in the carrying out of the full method as previously described (*loc. cit.*). This check was, however, employed in one case—that of the strong cyanide solution—which gave the CNO as 0.014 and 0.015 N. as against the 0.013 and 0.015 already quoted.

It is obvious from the above results that the two determinations give equal results if NH₄ salts be not present in the original sample. But still it seems doubtful whether this would be the case if the HCN were not quickly got rid of in the first boiling with excess of acid, as it is probable that prolonged treatment of this sort would hydrolyse some of the cyanide with production of NH₃. As some of the analytical methods that have been recommended by others would be spoiled by such an action, it appeared worth while to test the question. For this purpose the same quantity of the same normal cyanide solution as was used previously was boiled for about three hours with excess of sulphuric

acid in a flask provided with a reflux condenser, and the ammonia was then determined. The quantity obtained, if assumed due only to the decomposition of cyanate, would make the latter 0.030 N., whereas the true value was 0.014 N., as already shown. It seems, therefore, that there is some danger in such prolonged heating, though it is certainly negligible when the boiling is effected quickly in an open vessel, as is our practice.

University of Melbourne.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, December 21st, 1905.

Prof. R. MELDOLA, F.R.S., President, in the Chair.

MESSRS. P. W. Robertson, Arthur Slator, J. I. Scott, and E. Towyn Jones were formally admitted Fellows of the Society.

Certificates were read for the first time in favour of Messrs. Thos. Going Stoney Bogue, 5, Kenilworth Square, Dublin; Stephen Lewis Courtauld, B.A., Bocking Place, Braintree; Henry Carlyle Irving, B.A., 14, Heath Hurst Road, Hampstead, N.W.; John McDowall, Girdstingwood, Kirkcudbright, Scotland; John Parkin, M.A., Blaithwaite, Carlisle; William Hughes Perkins, B.Sc., Harpur Hill, near Buxton; Philip Foale Rowsell, Nutbrook, Exmouth; John William Yates, B.Sc., 71, North Street, Hugglescote, near Leicester.

A certificate has been authorised by the Council for presentation to ballot under By-law I. (3) in favour of Hardolph Wastenays, of Brisbane, Queensland.

Of the following papers, those marked * were read:—

*218. "*Aso-derivatives from Methyl- α -naphthocoumarin.*" By JOHN THEODORE HEWITT and HERBERT VICTOR MITCHELL.

The methyl- α -naphthocoumarin, obtained by the condensation of α -naphthol and ethyl acetoacetate, when dissolved in hot caustic alkalis, yields alkali coumarinates, and if these solutions are cooled and treated with the solution of a diazonium salt, coupling takes place in the usual manner. On acidifying the solution, the corresponding azocoumarin is precipitated, and in none of the four cases examined has the formation of an azocoumarinic acid been observed.

The following substances have been prepared:—*Benzene-asomethylnaphthocoumarin* (m. p. 207°); *o-nitrobenzene-asomethylnaphthocoumarin* (m. p. 268°); *m-nitrobenzene-asomethylnaphthocoumarin* (m. p. 239°); *p-nitrobenzene-asomethylnaphthocoumarin* (m. p. 270–271°). The last of these compounds is remarkable in giving an intense blue coloration in alkaline solution, the alkaline salts derived from the other compounds being orange or red in aqueous solution.

*219. "*The Preparation and Reactions of Benzoyl Nitrate.*" By FRANCIS ERNEST NICHOLS.

Lachowicz showed (*Ber.*, 1884, xvii., 1281) that when benzoyl chloride or other chlorides of the mono- and dibasic organic acids interact with silver or other metallic nitrates, decomposition into oxides of nitrogen and the corresponding organic anhydrides takes place.

If, however, the interaction of benzoyl chloride with silver nitrate is effected at much lower temperatures, oxides of nitrogen are not evolved, and benzoyl nitrate is formed, $C_6H_5 \cdot COCl + Ag \cdot O \cdot NO_2 = C_6H_5 \cdot CO \cdot O \cdot NO_2 + AgCl$.

The product is a light yellow oil which, if carefully warmed, decomposes quantitatively into benzoic anhydride and oxides of nitrogen, but if heated with a free flame this change sets in with explosive violence, the yield of anhydride being much diminished.

Benzoyl nitrate is very rapidly attacked by moisture with

the formation of benzoic and nitric acids. When kept at low temperature either by itself or dissolved in an inert solvent, an interesting change into *m*-nitrobenzoic acid takes place without the formation of either the *o*- or *p*-acid.

Between 0° and -15°, ethyl alcohol and the nitrate give ethyl nitrate, benzene and toluene are converted into their mononitro-derivatives, and phenetole and phenol undergo nitration quantitatively, the product from the former being apparently *o*-nitrophenetole. Methylaniline is nearly quantitatively converted into phenylmethyl-nitramine, and in all these reactions benzoic acid is simultaneously formed. Aniline, however, gives aniline nitrate and benzanilide. From these observations, it seems probable that benzoyl nitrate will prove to be generally applicable in effecting nitration at low temperatures and in the absence of water.

*220. "The Supposed Identity of Dihydrolaurolene and of Dihydroisolurolene with 1:1-Dimethylhexahydrobenzene." By ARTHUR WILLIAM CROSSLEY and NORA RENOUF.

The hydrocarbons dihydrolaurolene and dihydroisolurolene have been prepared according to the directions given by Zelinsky and Lepeschkin (*Annalen*, 1901, cccxix., 303), and their properties carefully compared with those of 1:1-dimethylhexahydrobenzene (*Trans.*, 1905, lxxvii., 1488). The above-mentioned authors supposed that these three substances were one and the same, but this is not so, for neither dihydrolaurolene nor dihydroisolurolene is identical with 1:1-dimethylhexahydrobenzene.

The constitution of dihydrolaurolene is still doubtful, but dihydroisolurolene has been proved conclusively to be a pentamethylene derivative, and is 1:1:2-trimethylcyclopentane.

*221. "The Diazo-derivatives of 1:5- and 1:8-Benzenesulphonylnaphthylenediamines." By GILBERT THOMAS MORGAN and FRANCES MARY GORE MICKLETHWAIT.

The behaviour of the benzenesulphonyl derivatives of the 1:5- and 1:8-naphthylenediamines towards nitrous acid has been studied in order to ascertain whether these acylated heteronuclear diamines are capable of yielding diazoimides, and if so to compare the products with the reactive yellow arylsulphonylparadiazoidimides and the inert colourless acylorhodiazoimides.

Benzenesulphonyl-1:8-naphthylenediamine (m. p. 166°) obtained by reducing *benzenesulphonyl-8-nitro-2-naphthylamine* (m. p. 194°), yields a soluble diazonium salt which, by the action of aqueous sodium acetate, gives rise to *benzenesulphonyl-1:8-naphthylenediazoimide*, $C_{10}H_6[N_3] \cdot SO_2 \cdot C_6H_5$; this product is a well-defined, crystalline, yellow diazoanhydride comparable in every respect with the *paradiazoidimides* already described by the authors.

This result indicates that the property of yielding these coloured diazoimides is not confined to the *p*-diamine series, and that the characteristic physical and chemical properties of the products do not necessarily arise from the possession of a *para*quinonoid configuration.

as-Benzenesulphonyl-N-methyl-1:8-naphthylenediamine and *benzenesulphonyl-1:5-naphthylenediamine* furnish diazonium salts which are not appreciably affected by aqueous sodium acetate.

*222. "Further Experiments on a New Method of Determining Molecular Weights." By PHILIP BLACKMAN.

When working with solvents of high boiling-point, the operations (*Trans.*, 1905, lxxvii., 1474) must be carried out under reduced pressure, this reduction being effected by means of a filter-pump.

To prevent ejection of liquid or mixing, due to violent boiling, two bulbs, instead of one, should be introduced into each limb of the inverted U-piece.

Most of the vapours condense in the bulbs; when a considerable quantity has thus collected in the bulbs, the volumes of the solutions in the test-tubes are observed, care being taken that the liquid in the bulbs does not flow back into the test-tubes during the reading. The liquid is then

allowed to return into the tubes, the boiling is repeated, and a reading again taken. This process is repeated as often as is convenient.

A condenser should not be used. Instead of platinum wire, broken pieces of glass were employed. The method works best with volatile solvents, such as acetone (b. p. 56.5–57°), alcohol (96 per cent), and benzene (b. p. 80–82°).

*223. "Studies in Fermentation. The Chemical Dynamics of Alcoholic Fermentation by Yeast." By ARTHUR SLATOR.

The chief results obtained may be summarised as follows:—

1. In the study of the rate of alcoholic fermentation, many complications are eliminated by measuring the velocity over very small ranges of the reaction.

2. The change in pressure due to evolution of carbon dioxide is a convenient and sensitive method of measuring this velocity.

3. The rate of fermentation of dextrose is proportional to the concentration of yeast over a wide range of concentrations.

4. The rate is almost independent of the concentration of sugar except in very dilute solutions. The influence of this concentration is never so great that the velocity is proportional to the concentration of the sugar; the reaction is therefore never one of the first order with regard to the sugar.

5. The temperature-coefficient of the reaction is large, and varies with the temperature. $V_{15}/V_5 = 5.6$, $V_{40}/V_{30} = 1.6$, and intermediate values are obtained between these temperatures. The temperature quotient for 5° from 5° to 40° forms a series of numbers which seem to be characteristic of the enzyme zymase.

6. The initial rates of fermentation of dextrose, lævulose, sucrose, and maltose are in the ratio 1:0.92:1.05:0.9.

7. The temperature-coefficient of the reaction inhibited by "poisons" is the same as that of the original reaction.

8. It is improbable that any but small quantities of sugar go through the intermediate step of lactic acid in fermentation.

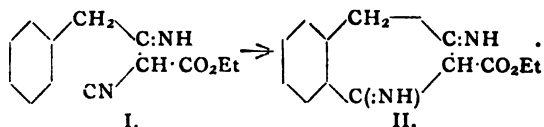
9. These results indicate that the reaction which is measured in these experiments is the slow decomposition of a compound produced by the interaction of the enzyme and the sugar.

*224. "Some New Platinocyanides." By LEONARD ANGELO LEVY and HENRY ARNOTT SISSON.

The authors described and gave the results of the analysis of hydrazine and hydroxylamine platinocyanides. These salts form unstable hydrates, which are easily produced by slight changes in temperature. The alteration in the state of hydration is accompanied by striking colour changes.

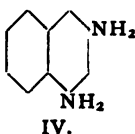
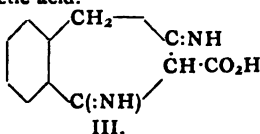
*225. "An Intramolecular Change Leading to the Formation of Naphthalene Derivatives." By ERNEST FRANCIS JOSEPH ATKINSON and JOCELYN FIELD THORPE.

Ethyl sodiocyanoacetate condenses with benzyl cyanide to form *ethyl α-cyano-β-imino-γ-phenyl-n-butyrate*, $Ph \cdot CH_2 \cdot C:(NH) \cdot CH(CN) \cdot CO_2Et$, a colourless substance crystallising from alcohol in large prisms melting at 125°; its constitution is proved by the fact that on hydrolysis with alcoholic potash it yields phenylacetic and malonic acids. The new imino-ester (I.), when treated with an equal weight of cold concentrated sulphuric acid, forms an intense green solution, which, when diluted with water and rendered alkaline with ammonia, gives *ethyl 1:3-diamino-naphthalene-2-carboxylate* (II.), forming intense yellow prisms melting at 104°.



The hydrochloride forms colourless needles. Ethyl 1:3-diaminonaphthalene-2-carboxylate, on treatment with

methyl-alcoholic potash, gives a potassium salt from an aqueous solution of which 1:3-diaminonaphthalene-2-carboxylic acid (III.) is precipitated on acidifying with acetic acid.



The acid forms slightly coloured needles from warm water, which decomposes at about 85°, and when heated at this temperature until the evolution of carbon dioxide has ceased are transformed into 1:3-naphthlenediamine (IV.) which, when re-crystallised from dilute alcohol, forms small plates melting at 96°.

The diacetyl derivative forms needles from glacial acetic acid melting at 263°.

DISCUSSION.

In reply to questions from Drs. Forster and Morgan, Dr. THORPE said that the reaction with cold sulphuric acid proceeded very rapidly, and was complete after about one minute, the yield being practically quantitative. Hitherto the yield of ethyl α-cyano-β-imino-γ-phenyl-*n*-butyrate only amounted to 25 per cent, but by slightly altering the conditions of the experiment it was hoped that this amount would be largely increased. The temperature at which the condensation was carried out greatly influenced the quantity of the condensation product formed.

226. "The Relation of Position Isomerism to Optical Activity. V. The Rotation of the Menthyl Esters of the Isomeric Dibromobenzoic Acids." By JULIUS BEREND COHEN and ISRAEL HYMAN ZORTMAN.

In continuation of previous investigations on this subject (*Trans.*, 1903, lxxxiii., 1213; 1904, lxxxv., 1262, 1271; 1905, lxxxvii., 1190), the present communication contains an account of certain physical constants, including the molecular rotations of the six isomeric menthyl dibromobenzoates. With the exception of the 2:6-ester, the effect on the rotation of the substitution of two bromine atoms is generally less than that of two chlorine atoms, or one chlorine and one bromine, in the same positions. The magnitude of the deviation, beginning with the ester of smallest rotation, is 2:6; 2:3; 2:5; 2:4; 3:5; 3:4; phenyl. The order is the same as that of the dichloro- and chlorobromobenzoates with the exception of the 3:4- and 3:5-esters, the positions of which in the present case are reversed. The influence of the ortho-bromine atom in depressing the rotation is very clearly indicated in all cases, but most strikingly in that of the 2:6-ester, which shows a rotation of $[M]_D^{20} - 19.5^\circ$ compared with the next lowest (2:3), which gives $[M]_D^{20} - 173.2^\circ$, or with menthyl benzoate, in which $[M]_D^{20}$ is -236.3° .

227. "Some Derivatives of Naphthoylbenzoic Acid and of Naphthacenequinone." By JAN QUILLER ORCHARDSON and CHARLES WEIZMANN.

The authors have investigated a series of condensations which have resulted in the preparation of substitution derivatives of naphthoylbenzoic acid, and from these, by the action of concentrated sulphuric acid, they obtained the corresponding substituted naphthacenequinones.

228. "Ethyl β-Naphthoylacetate." By CHARLES WEIZMANN and ERNEST BASIL FALKNER.

β-Naphthoic acid was converted into β-naphthoyl chloride, $C_{10}H_7COCl$, by treatment with phosphorus pentachloride, and this chloride was then allowed to react with the sodium derivative of ethyl acetoacetate. The ethyl β-naphthoylacetate, $C_{10}H_7COCH_2CO_2Et$, melted at 57°, and when digested with ammonia and ammonium chloride yielded ethyl β-naphthoylacetate, which melted at 34°, and gave rise to a hydrazone (m. p. 95°).

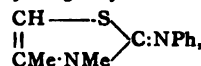
229. "Contributions to the Chemistry of the Amidines. 2-Aminothiazoles and 2-Imino-2:3-dihydrothiazoles. 2-

Iminotetrathiazoles and 2-Amino-4:5-dihydrothiazoles." By GEORGE YOUNG and SAMUEL IRWIN CROOKES.

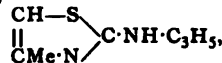
It was shown that the alkylation of amidines belonging to the thiazole group which have one of the nitrogen atoms and the carbon atom of the group $-N:C:N-$, forming part of a closed chain, whilst the other nitrogen atom lies outside the cyclic nucleus, leads to the formation of derivatives in which the alkyl group is attached to the nitrogen atom of the nucleus, except when the ring is already partially reduced and an aryl group is attached to the nitrogen atom of the side-chain, to which, in this case, the alkyl group becomes attached. Methylation of an amidine takes place by addition of methyl iodide and subsequent elimination of the hydrogen haloid.

The following substances were described:—

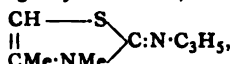
Acetyl-2-anilino-4-methylthiazole, $C_{10}H_9N_2S$ Ac, forms clusters of soft, white needles (m. p. 114.5°); 2-phenylimino-3:4-dimethyl-2:3-dihydrothiazole,—



separates in small, white crystals (m. p. 65–66°), and forms a platinichloride (m. p. 189–190°). 2-Allylimino-4-methylthiazole,—

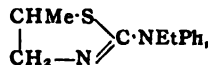


crystallises in long, white needles (m. p. 40–41°), and forms an acetyl derivative (m. p. 36–37°). 2-Allylimino-3:4-dimethyl-2:3-dihydrothiazole,—



is a slightly red, viscid oil which forms a hydriodide (m. p. 116–117°). The silver derivative of 2-acetyl-2-methyl-2:3-dihydrothiazole yields, on methylation, 2-acetyl-2-methyl-2:3-dimethyl-2:3-dihydrothiazole (m. p. 113°); 2-methylamino-4-methylthiazole (m. p. 64°) yields, on methylation, 2-methyl-2-methyl-2:3-dimethyl-2:3-dihydrothiazole hydriodide (m. p. 164°).

2-Phenylimino-5-methyltetrahydrothiazole forms a silver derivative decomposing at 130° and an acetyl derivative melting at 47°; 2-phenyl-methylamino-5-methyl-4:5-dihydrothiazole picrate forms soft yellow needles (m. p. 114–115°); 2-phenylethylamino-5-methyl-4:5-dihydrothiazole,—



is oily, and forms a platinichloride decomposing at 156°.

A similar series of compounds, in which the *p*- and *o*-tolyl groups replace the phenyl group of the foregoing substances, has also been prepared.

When successively oxidised and hydrolysed, the 2-arylalkylamino-5-methyl-4:5-dihydrothiazoles yields β-methyltaurine, which melts at 284–285°, and the corresponding arylalkylamine.

230. "The Action of Water on Diazo-salts." By JOHN CANNELL CANE and GEORGE MARSHALL NORMAN.

The authors have extended the work already briefly described (*Proc.*, 1905, xxi., 206) to the examination of the diazo-salts from 2:4-dibromoaniline and dibromo-*p*-toluidine ($CH_3NH_2:Br:Br=1:4:3:5$), and in each case were successful in obtaining the corresponding substituted phenols. The results of a series of researches carried out during the past four years by the author and his assistants have been summarised.

231. "Note on the Atomic Weight of Nitrogen." By ALEXANDER SCOTT.

By the titration of ammonium bromide against silver, the author (*Trans.*, 1901, lxxix., 154) obtained results which differed notably from those of Stas, although the

values from the chloride seemed to agree somewhat better. These values were:—

	Stas.	Scott.
NH ₄ Cl	= 53'532	53'516
N	= 14'045	14'029
NH ₄ Br	= 98'032	97'995
N	= 14'047	14'010

Stas' values for the atomic weight of nitrogen agree with one another, whilst those of the author do not. These values depend, however, on the correctness of the atomic weights taken for chlorine and for bromine, which were assumed to be 35'457 for chlorine and 79'955 for bromine.

By the recent determinations of T. W. Richards and R. C. Wells ("A Revision of the Atomic Weights of Sodium and Chlorine," 1905), published by the Carnegie Institution of Washington, the want of agreement in the author's values has been completely cleared up, and the discrepancy (to an equal extent) transferred to the determinations made by Stas. They prove that Cl = 35'473.

The work of the author was criticised last year by T. W. Richards (*Proc. Am. Phil. Soc.*, 1904, xliii., 116), who maintained that 14'01 was too low for the atomic weight of nitrogen, and at the end of his paper he states that:—"It seems probable that the atomic weight of nitrogen is not less than 14'02 and not over 14'04, probably being nearer to the latter value than to the former."

The physical work of Lord Rayleigh and of Leduc on this problem as well as the chemical and physical work of Guye and his co-workers, and of R. W. Gray (*Trans.*, 1905, lxxxvii., 1601) all render the conclusions of Richards quite untenable. It is most interesting that the author's critic should, by his own work, have brought the values of the author, which are cited above, completely into line with those of other recent workers. Using 35'473 instead of 35'457 for the atomic weight of chlorine as above, the author's values for the atomic weight of nitrogen become 14'013 from the chloride and 14'010 from the bromide. When it is remembered that only one gravimetric and two volumetric determinations were made with the chloride, closer agreement could not be expected, and the value 14'010 deduced from the large number of experiments made with the bromide seems completely established.

With regard to the suggestion that the low value found by the author for the bromide was due to his hydrobromic acid containing chlorine, the results given in his paper on the preparation of pure hydrobromic acid (*Trans.*, 1900, lxvii., 651) are a sufficient answer. The bromine used was purified by the method given by Stas, by solution in potassium bromide with the addition of zinc oxide. It ought to have been stated that the silver used in Experiment VI.(b) (*Trans.*, 1901, lxxix., 152) was cleaned by acid, as recommended by Richards, after fusion on lime and before it was heated in hydrogen.

232. "The Solubility of Zinc Hydroxide in Alkalis."

By JAMES MOIR.

When the precipitate of zinc hydroxide dissolves in excess of caustic alkali, the phenomenon is essentially an equilibrium between the alkali and zincic acid, and this equilibrium may be reached from both sides:—(1) addition of excess of zinc hydroxide to caustic alkali, and (2) dilution with water of a strong solution previously saturated with zinc hydroxide. In the latter case, some of the hydroxide is re-precipitated and an equilibrium is slowly reached, which, as in the first case, depends solely on the concentration of the free alkali.

Observations have been made on both lines over the range from 7 N to 0'01 N alkali, below which the amount of zinc dissolved is insignificant, being little greater than that due to the solubility of zinc hydroxide in pure water. In addition, the author has succeeded in showing that the experimental results can be deduced from the ordinary assumptions of the ionic theory; so that it follows that the solubility of zinc hydroxide in alkali depends solely on the concentration of hydroxidion in the solution.

It is also shown that no definite chemical compounds (such as ZnO,8KOH) exist, since the curve obtained by

plotting the results is approximately a parabola, and its tangent never passes through the origin.

A practical result of the investigation is to show that in working cyanide solutions scarcely any of the zinc is present as zincate, but nearly all is zincocyanide.

The formula, giving the dissolved zinc in terms of the concentration of alkali, is $y = 0'004x \left(\frac{79x+6}{x+2} \right)$; where y and x are expressed in grm.-molecules.

The following list gives a comparison of calculated values of y with those obtained by interpolation from the experimental curves:—

	$x =$	y Calculated =	y Observed =
1. 7'5 N NaOH ..	7'5	1'89	1'692
2. 5'5 N KOH ..	5'5	1'291	1'36
3. 2'5 N NaOH ..	2'5	0'452	0'48
4. 2'0 N KOH ..	2'0	0'328	0'32
5. 1'5 N KOH ..	1'5	0'2033	0'21
6. 1'3 N NaOH ..	1'3	0'171	0'17
7. 1 N KOH ..	1	0'1132	0'110
8. 0'5 N NaOH ..	0'5	0'0364	0'040
9. 0'1 N NaOH ..	0'1	0'00265	0'0035
10. 0'05 N NaOH ..	0'05	0'00097	0'0010
11. 0'01 N NaOH ..	0'01	0'000135	0'0002

The theoretical results are usually a little too low, which points to a small error in the value of the constants assumed; but on the whole the agreement between theory and practice is as good as could be expected, considering that the experiments were not made at absolutely constant temperature. This agreement cannot be fortuitous, and vindicates the correctness of the assumptions made at the beginning regarding the constitution of zincate solutions and the applicability of the ionic theory. They show also that the whole phenomenon is not one of the formation of definite chemical compounds, but is purely conditioned by the strength of the alkali.

Some of the experimental results are abnormal, especially those obtained by diluting a strong saturated zincate solution. This effect is due to the persistence of the state of supersaturation. The reactions $K_2ZnO_2 + H_2O \rightarrow KHZnO_2 + KOH \rightarrow H_2ZnO_2 + 2KOH$ have evidently slow velocities, nevertheless the hydrolysis is eventually very complete, and ultimately the same result is obtained as if alkali of the final concentration had been treated with excess of zinc hydroxide. Zinc hydroxide, acting as zincic acid, has therefore a different constant of dissociation from its ordinary basic one, and, in fact, the constant of the equation $(H^+)(HZnO_2) = K.(H_2ZnO_2)$ is rather less than that of water, a fact which accounts for the extensive hydrolysis of the salts of zincic acid.

233. "The Slow Combustion of Carbon Disulphide."

By NORMAN SMITH.

Experiments have been carried out in order to determine the composition of the reddish-brown deposit formed when carbon disulphide and oxygen are passed through a heated tube (compare Turpin, *Brit. Assoc. Reports*, 1890, 776; Dixon and Russell, *Trans.*, 1899, lxxv., 603). The deposit consists chiefly of an acidic compound $C_{16}H_6O_4S_8$. Another acidic substance or substances containing less carbon and more sulphur than the foregoing product, and very small quantities of free carbon and sulphur, are also found to be present. The silver and ammonium salts of the compound $C_{16}H_6O_4S_8$ have been prepared. The gaseous products consist chiefly of sulphur dioxide with small amounts of carbon dioxide.

An International Exhibition at Antwerp is announced for the months of April and May, 1906. Chemistry and Pharmacy will occupy very important places. Manufacturers are strongly advised to take part in this Exhibition, which is under Official patronage, and under the Honorary Presidentship of S. A. R. Madame la Comtesse de Flandres. Further particulars can be obtained from the Secretary, 26, Rue d'Arenberg, Antwerp (The Royal Artistic Club).

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances de l'Académie des Sciences. Vol. ccli., No. 24, December 11, 1905.

Distillation of Gold, of the Alloys of Gold with Copper and with Tin, and on a New Method of Preparation of Purple of Cassius.—Henri Moissan. *Distillation of Gold.*—Following the experience derived from the distillation of copper, the author by varying the time of action of a current of 500 ampères under 110 volts, acting on 150 grms. of pure gold, succeeded in distilling as much as 20 grms. of metal. He describes at length the residue of gold left in the crucible, which is found to be free from lime. The surface of the tube of copper, placed above the crucible and cooled by a current of water, is covered with a layer which, on examination with a lens, resembles that described in a previous note. Close to the tube were found very small yellow cubic crystals accompanied by a small quantity of lime and graphite. The vapour condensing on a bell-glass gave a beautiful purple deposit, consisting of a mixture of lime and distilled gold. The author then repeated these experiments in a carbon tube, the metal being placed in a graphite boat. Several small crystals of gold were found amongst a large number of metallic spherules, and the residue was covered by a film of graphite as above. The author concludes that the distillation of gold is more difficult than that of copper, and that it takes longer, and that gold is already volatile at the temperature of fusion of lime. The chemical properties of distilled gold are the same as those of hammered or molten metal reduced to a fine powder. Aqua regia and chlorine water attack it according to the degree of tenuity of the specimen. This is equally true for the action of fluorine or sulphuric acid and potassium permanganate. *Distillation of Alloys of Copper and Gold.*—Roberts-Austen has given the curve of fusibility of these alloys, and has shown that they form homogeneous solutions in all proportions up to a temperature of 1064°. The author has prepared an alloy containing 10 per cent of gold, and after heating 100 grms. for four minutes with a current of 500 ampères under 70 volts, obtained as residue 77 grms. Copper, 89.02; gold, 11.33 per cent. Another experiment with 39 grms. of 10 per cent gold gave, after five minutes,—Gold, 10.72; copper, 89.09. With a 50 per cent alloy he obtained:—(a) Gold, 50.22; copper, 49. (b) Gold, 57.02; copper, 42.81. He concludes that copper is certainly more volatile than gold. *Alloy of Gold and Tin.*—In this case numerous experiments show that tin distils more rapidly than gold, the actual figures being:—200 grms. of an alloy, containing 40 per cent of gold, heated for three minutes in a current of 500 ampères under 70 volts, left 185 grms. of residue, consisting of:—(a) Gold, 41.08; tin, 59.72; and for four minutes 149 grms. of residue—(b) Gold, 45.9; tin, 53.88. *Purple of Cassius.*—The purple powder obtained on the condensing tube and on the bell-glass is of the same colour as the areola round the little globule of gold condensed on the lime of the electric furnace some distance from the crucible, and due to the formation of anhydrous calcium oxide coloured by gold. It is, then, a new purple prepared by the volatilisation of gold, and the condensation of its vapour on the lime. When the vapour from the alloy of gold and tin is given off, the tin burns to oxide, and is mixed with the vapour of gold, which is unaffected by air; the substance formed has the composition— SnO_2 , 49.15; CaO , 36.93; Au , 9.9. This composition varies in each experiment and depends on the quantity of lime and metals volatilised in the electric furnace. But it has the properties of the purple of Cassius, and on being freed from the lime by hydrochloric acid gives the well known colour. By

volatilising gold with different metallic oxides, the author has been able to vary the purple, and his experiments confirm Debray's ideas on the constitution of the purple of Cassius which showed that it had no definite composition, being merely a lacquer of tin coloured by a fine layer of gold. The presence of graphite is due to the fact that gold at high temperatures dissolves it, and the author has performed experiments to test this by boiling gold in a carbon crucible and then plunging it into cold water. On examination of the cooled contents, crystals of graphite were found in it. *Conclusions.*—Gold distils readily in the electric furnace at a temperature above that of copper and below that of lime. In the alloys with copper and tin, these metals distil before the gold, and from the latter alloy purple of Cassius is obtained, which can be varied in colour by using different oxides.

Two Iodo-mercurates of Lithium.—A. Duboin.

A New Compound: Fluoride of Bromine, BrF_3 .—Paul Lebeau.

MEETINGS FOR THE WEEK.

TUESDAY, 16th.—Royal Institution, 5. "Impressions of Travel in China and the Far East," by Prof. Edward H. Parker, M.A.

WEDNESDAY, 17th.—Society of Arts, 8. "The Scientific Aspects of Voice Development," by Dr. W. A. Aiken. Microscopical, 8. Annual Address of the President—"The Life and Works of Bernard Renault."

THURSDAY, 18th.—Royal Institution, 5. "Shakespeare," by the Rev. Canon Henry Charles Beeching, M.A., &c. Society of Arts, 4.30. "The City of Calcutta," by Charles E. Buckland.

Society of Arts, 8. (Howard Lecture). "High Speed Electrical Machinery, with special reference to Steam Turbine Machines," by Prof. Silvanus Thompson, F.R.S.

Chemical 8.30. "Refractive Indices of Crystallising Solutions, with especial reference to the Passage from the Meta-stable to the Labile Condition," by H. A. Miers and F. Isaac. "Determination of Available Plant Food in Soils by the Use of Weak Acid Solvents," by A. D. Hall and A. Amos. "Action of Ammonia and Amines on Diazobenzene Picrate," by O. Silberrad and G. Rotter. "Preparation of β -bis-Triazobenzene," by O. Silberrad and R. J. Smart. "Gradual Decomposition of Ethyl Diazacetate," by O. Silberrad and C. S. Roy. "Studies on Nitrogen Iodide—Part III., Action of Methyl and Benzyl Iodides," by O. Silberrad and B. J. Smart. "Silicon Researches—Part X., Silicon Thiocyanate," by J. E. Reynolds. "Relations between Absorption Spectra and Chemical Constitution—Part I., The Chemical Reactivity of the Carbonyl Group; Part II., Quinones and α -Diketones," by E. C. C. Baly and A. W. Stewart; Part III., "The Nitroanilines and the Nitrophenols," by E. C. C. Baly, W. H. Edwards, and A. W. Stewart. "Halogen Derivatives of Substituted Oxamides," by F. D. Chattaway and W. H. Lewis. "Effect of Constitution on the Rotatory Power of Optically Active Nitrogen Compounds" (Part I.), by Miss M. B. Thomas and H. O. Jones. "Menthyl Benzene Sulphonate and Menthyl- β -naphthalene Sulphonate," by T. S. Patterson and J. Frew. "Apparatus for the Continuous Extraction of Liquids with Ether," by R. S. Bowman. "Action of Bromine on Benzeneazo-o-nitrophenol," by J. T. Hewitt and N. Walker. "Some Reactions and New Compounds of Fluorine" (Part I.), by E. B. R. Priceaux. "Contributions to the Chemistry of the Rare Earths" (Part I.), by M. Raposo. "Synthesis of Aldehydes by Grignard's Reaction," by G. W. M. Williams. "Condensat on of Dimethyldihydroresorcin and of Chloroketodimethyltetrahydrobenzene with Primary Amines—Part I., Monoamines, Ammonia, Aniline, and p Toluidine," by F. Haas.

FRIDAY, 19th.—Royal Institution, 9. "Some Applications of the Theory of Electric Discharge to Spectroscopy," by Prof. J. J. Thomson, F.R.S., &c.

SATURDAY, 20th.—Royal Institution, 3. "The Church in France," by J. E. C. Bodley.

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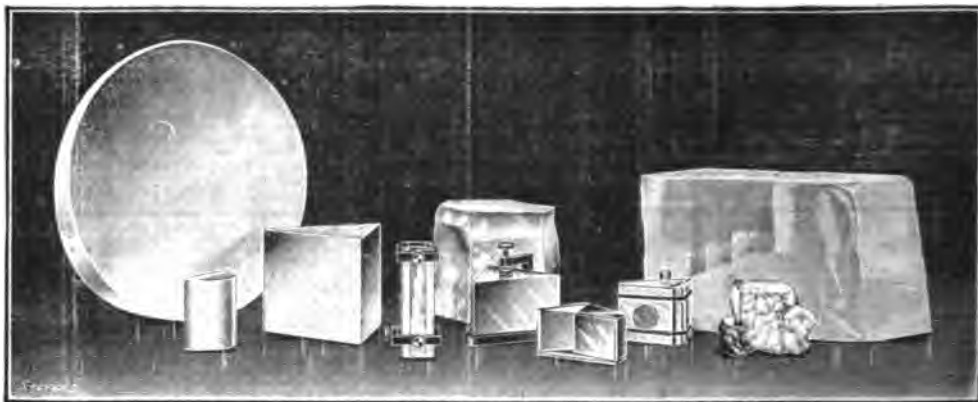
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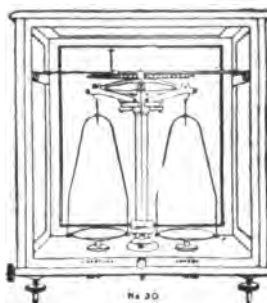
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THE CHEMICAL NEWS.

VOL. XCIII., No. 2408.

NEW RESEARCHES ON THE ATOMIC WEIGHT OF NITROGEN.*

By PHILIPPE A. GUYE.

(Continued from p. 14).

Volumetric Analysis.

A GLASS vessel containing an iron spiral, analogous to that used for the gravimetric analysis, is connected with a mercury manometer, in which the mercury is brought up to a certain level so as to operate at constant volume.

The vessel after being filled with nitrous oxide is surrounded with ice, and the initial pressure registered. The ice is then removed, and the oxide decomposed by raising the spiral electrically to a red-heat. After cooling, the pressure of the nitrogen is reduced to 0°, a slightly higher pressure than the initial one. After all corrections for the compressibility of the gases and the difference of temperature of the space required have been made, the excess of the pressure obtained above the initial pressure of 760 m.m. of mercury was found equal to the following values:—

TABLE XIV.

Pressures reduced to 0°.		Increase for 760 m.m.
N ₂ O.	N ₂ .	
745·93	751·37	5·60
764·90	770·29	5·30
767·72	773·26	5·43
762·97	768·49	5·45

Mean.. .. 5·44

It has been found that the mean 5·44 should be subjected to a small correction, from the fact of the diminution of capacity of the glass vessel due to the transformation of part of the iron of the spiral into iron oxide. This correction brings the number from 5·44 to 5·21 m.m.

The ratio of the volumes of the two gases N₂ : N₂O is therefore—

$$\frac{N_2}{N_2O} = \frac{765.21}{760.00} = 1.00686.$$

The extreme values are—

$$1.00707 \text{ and } 1.00667,$$

the difference being 4/10000 only, corresponding probably to a slight uncertainty of 1/5000 on the mean.

Adopting for the densities of the gases, nitrogen and nitrous oxide, the mean of all the found values, we have deduced that:—

1 litre N ₂ O, weighing	1.97772
Contains 1.0068 litre N ₂ , weighing	1.25045 ×
1.00686	1.25903

And, by difference, a weight of oxygen 0.71869

Whence—

$$N = \frac{16}{2} \frac{1.25903}{0.71869} = 14.015.$$

Allowing, with Jaquero and Bogdan, for an uncertainty of 1/10000 on the densities of N₂ and N₂O, and of 2/10000 on the volume ratio, the error on the atomic weight of nitrogen would be 7/10000 if these causes of error act in the same direction, and 1.7/10000 if they compensate as well as possible, giving as mean 4.4/10000.

The possible error ΔN resulting on the atomic weight of nitrogen will be—

$$\Delta N = \pm \frac{14 \times 4.4}{10000} = \pm 0.0067.$$

The two methods—gravimetric and volumetric analysis—afford, therefore, under the given conditions, sufficient accuracy to determine the second decimal of the atomic weight of nitrogen.

The result to which we have been led has also been recently confirmed by the complete analysis of a second oxide of nitrogen, the dioxide by Gray. In his first notice he announced that he had effected the complete analysis of this gas by fixing the oxygen with nickel, and condensing the nitrogen upon charcoal cooled to the temperature of liquid air. This process therefore presents great similarity to those employed in my laboratory for the analysis of the suboxide. The mean of six analyses led Gray to the value—

$$N = 14.006,$$

which he explains as "probably a little too low."

Placing together the physico-chemical and gravimetric methods, we are led to the following recapitulation:—

TABLE XV.—General Recapitulation.

A. Physico-chemical Methods.

Ratio	N ₂ : O ₂ (6 values)	N.
"	N ₂ : CO (6 values)	14.009
"	N ₂ O : CO ₂ (2 values)	14.006
"	NO : O ₂ (2 values)	14.007
		14.008
Physico-chemical mean (4 ratios)		14.008

B. Gravimetric Methods.

Analysis by weight of N ₂ O (5 expts.)	14.007 (G. and B.)
" by volume of N ₂ O (4 expts.)	14.015 (J. and B.)
" by weight of NO (6 expts.)	14.006 Gray

Gravimetric mean (3 ratios)	14.009
General mean	14.0085
Most probably	14.009

In the face of such complete agreement between the means of the two groups of methods, which only make use of the direct ratios to oxygen, or to compounds relatively rich in oxygen, it is certainly logical to renounce the old value N = 14.04 or 14.055 of Stas' experiments, in favour of either the mean—

$$N = 14.009$$

or the round number—

$$N = 14.01,$$

which only differs from the preceding one by 1/14000.

One might even use the value N = 14.00 which many chemists have continued to use; the error thus committed is less than that recently discovered by Richards in the atomic weight of sodium.

V. Conclusions and Consequences.

Recent researches clearly point to the revision of the atomic weight of nitrogen in the direction which brings it almost to the value that Marignac, in 1843, considered as the most probable.

This revision carries with it an important consequence to which I would draw your attention.

If the methods employed by Stas, his forerunners, or his successors, do not admit to-day of sufficient accuracy to ascertain the second decimal of the atomic weight of nitrogen, it is none the less true that the mean of these numerous experiments approximates to N = 14.04; the extreme values of all the means are, according to Clarke (1897), N = 14.017 and N = 14.053. According to the laws of large numbers, this result is only compatible with an exact value, N = 14.009 or N = 14.01, if the atomic weights

* From the Bulletin de la Société Chimique de Paris, 1905.

considered known by the old methods are a little too high. I say a "little," because calculation shows that very slight errors in these atomic weights become considerably multiplied in arriving at nitrogen. An error of this kind can be demonstrated with sodium, the atomic weight of which, fixed by Stas at $\text{Na} = 23.043$, becomes, by the researches of Richards, $\text{Na} = 23.008$ (for $\text{Ag} = 107.93$) or $\text{Na} = 23.006$ (for $\text{Ag} = 107.92$). It is therefore of the highest interest that this work of revision should be carried out in a very complete manner.

It is to be foreseen that if one is led, in the course of these researches, to raise somewhat the values of certain atomic weights, as recently in the case of chlorine and iodine, other atomic weights will have to be lowered by nearly as much.

(To be continued.)

THE PHYSICAL AND CHEMICAL PROPERTIES OF IRON CARBONYL.*

By Sir JAMES DEWAR, M.A., Sc.D., LL.D., F.R.S.,
Jacksonian Professor in the University of Cambridge,

and
HUMPHREY OWEN JONES, M.A., D.Sc., Fellow of Clare College, and Jacksonian Demonstrator in the University of Cambridge.

(Concluded from p. 15).

Decomposition by Light.—Mond and Langer (*loc. cit.*) state that liquid iron carbonyl is rapidly decomposed by light, giving rise to a solid product and carbon monoxide. Determinations of the percentage of iron in the compound made by them indicated that the formula was $\text{Fe}_2(\text{CO})_7$, but the body was not obtained in a pure state. This behaviour to light constitutes the most striking difference between the carbonyls of iron and nickel, and was therefore examined more fully.

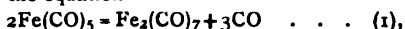
The decomposition was first investigated over mercury in a barometer tube. A weighed quantity of liquid iron carbonyl in a sealed bulb was introduced into a barometer and the bulb was then broken, after which the tube was exposed to light.

In the laboratory on bright days in February the decomposition was extremely slow, but on the same days in direct sunlight the decomposition was rapid and the evolution of gas was completed in a few hours. Exposure to the electric arc only induces the change very slowly, and a strong acetylene flame is almost without action. The volume of gas evolved (which was found to be pure carbon monoxide) was then measured and the solid collected. The solid was found to consist of beautiful, lustrous, hexagonal plates of an orange colour which, when pure, retained their lustre for a very long time on exposure to ordinary air, and indefinitely in dry air. If, however, the decomposition had not been completed and the solid was contaminated with traces of the liquid compound, rapid change occurred and the compound sometimes took fire.

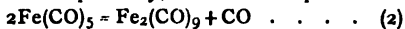
The following are two typical examples of experiments made in this way:—

1. 0.0905 grm. iron carbonyl gave 4.7 c.c. of carbon monoxide.
2. 0.2970 grm. iron carbonyl gave 18.4 c.c. of carbon monoxide.

According to the equation—



there should have been produced 15.5 c.c. and 50.9 c.c. of carbon monoxide respectively, whereas the equation—



requires 5.2 c.c. and 17 c.c. of carbon monoxide respectively.

It would, therefore, appear that the formula of the solid compound is $\text{Fe}_2(\text{CO})_9$ and not $\text{Fe}_2(\text{CO})_7$, and that the decomposition induced by light is represented by equation 2 above. This result was confirmed by carrying out the

decomposition in a large glass bulb out of contact with mercury.

A small sealed bulb filled with iron carbonyl was introduced into a large glass bulb, which was then sealed on to a glass tap, dried carefully, and exhausted with a Töpler pump. The small bulb was then broken and the whole exposed to light for some days, after which the carbon monoxide was pumped out and measured. In these experiments it was practically impossible to get the decomposition completed, since the bulb became covered with a deposit of the solid, which cut off the light.

The following are typical examples:—

- 0.6006 grm. of the liquid carbonyl gave 33.2 c.c. of carbon monoxide and left 0.501 grm. of solid.
- Theory requires 34.3 c.c. of gas and 0.557 grm. of solid.

- 0.5076 grm. gave 32 c.c. of gas. Theory requires 33.8.

Decomposition was incomplete, as shown by behaviour of solid, and by the fact that on admitting air to the carbon monoxide a deposit of iron oxide was produced. Another type of experiment also served to confirm the above conclusively:—

A carefully dried glass bulb of about 250 c.c. capacity was fitted with a small mercury manometer, thoroughly exhausted, and filled with dry iron carbonyl vapour, the pressure of which was measured at a definite temperature. The bulb was then exposed to light until no further change took place; after the bulb had been brought to the original temperature, the pressure was found to have been reduced to one-half its former value, as required by equation 2 above.

None of the specimens of the solid carbonyl prepared by either or the foregoing methods was found to be pure enough to give good results on analysis. The specimens prepared over mercury always retained small globules of mercury too small to be visible, but whose presence became evident during combustion, while the specimens prepared in glass bulbs were always contaminated with a little of the liquid, which either caused spontaneous combustion of the sample or decomposed quietly and left some oxidation product poor in carbon. The percentages of carbon monoxide obtained were too high for $\text{Fe}_2(\text{CO})_7$, 63.6 per cent, and too low for $\text{Fe}_2(\text{CO})_9$, 69.2 per cent; thus, for example, 65.0 and 66.5 per cent of carbon monoxide were obtained in two combustions.

We, therefore, examined the decomposition of the liquid in various solvents and succeeded in obtaining the compound in a pure state.

Iron pentacarbonyl dissolved in dry ether or petroleum ether and exposed to sunlight, undergoes rapid decomposition with evolution of carbon monoxide and formation of large reddish orange crystals of the solid carbonyl. To obtain the pure solid compound in quantity, the solution was sealed up in a dried and exhausted glass tube and exposed to light. Owing to the unavoidable exposure to air during the transference, a small amount of a precipitate of an oxidation product was sometimes produced. When a quantity of the solid carbonyl had been formed, the tube was opened (a considerable pressure of carbon monoxide was always produced), and the precipitate was removed easily by shaking the tube and pouring off the liquid, when the precipitate—which is very light and settles slowly—is poured off with the liquid, and the crystals remain behind. The crystals were then washed two or three times with the solvent and rapidly transferred to a desiccator containing sulphuric acid and solid paraffin. When prepared in this way they retain their lustre, and show no signs of change for a long time on exposure to air.

The percentage of carbon monoxide was then determined by combustion, using the same precautions as in the case of the liquid. The method of estimating the iron after decomposing the solid by heating in a sealed tube with bromine water was found unsatisfactory, and after trying several methods it was found that, if the compound were dropped slowly into pure nitric acid in a weighed

* A Paper read before the Royal Society, November 16, 1905.

crucible, complete decomposition took place, with effervescence and without loss of iron. The resulting liquid was then evaporated to dryness on a water-bath, the ferric nitrate decomposed by careful ignition, and the ferric oxide weighed.

The following results were obtained by these methods:—

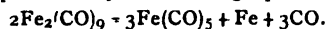
1. 0.2470 grm.	gave 0.2655 grm. CO ₂ .
2. 0.3178 " "	0.3417 " "
3. 0.1839 " "	0.1992 " "
4. 0.5791 " "	0.2534 grm. Fe ₂ O ₃ .
5. 0.3450 " "	0.1515 " "

Found—

	CO.	Fe.
1	68.4	—
2	68.4	—
3	68.9	—
4	—	30.6
5	—	30.7
Fe ₂ (CO) ₉ requires	63.60	36.40
Fe(CO) ₄ "	66.66	33.33
Fe ₂ (CO) ₉ "	69.20	30.80

The solid compound is, therefore, Fe₂(CO)₉, or *diferro-nonacarbonyl*. The solid prepared in this way forms large hexagonal plates, often 3 or 4 m.m. in breadth, but always rather thin. It is very sparingly soluble—in fact, practically insoluble—in ether, petroleum-ether, and benzene, but is slightly soluble in methylal, alcohol, and acetone, and much more soluble in pyridine to form a reddish solution. When dissolved the compound becomes much more sensitive to air and moisture, and deposits a reddish precipitate. We have not yet succeeded in re-crystallising the compound. The crystals are slightly dia-magnetic, but less so than the liquid. The density of the solid at 18° C. is 2.085, and its molecular volume is, therefore, about 174. If two gramme-molecules of solid Fe(CO)₅ were converted into Fe₂(CO)₉, then 256 volumes would become 174, or a contraction of about 33 per cent would ensue.

On heating solid iron carbonyl, as stated by Mond and Langer, liquid iron carbonyl is produced, and some solid, probably iron, is left. We find that this change takes place at about 100° C. Under a pressure of carbon monoxide up to 150 atmospheres, there is no rapid change below 95° C., but at this temperature the solid is completely converted into liquid iron carbonyl, though traces of a yellowish-brown solid are sometimes left. Quantitative experiments were made on the decomposition at 100° C. in a stream of hydrogen, and showed that the decomposition could be represented by the following equation:—



Liquid iron carbonyl, when exposed to light under a pressure of 75 to 125 atmospheres of carbon monoxide in the tube of a Cailliet pump, decomposes without any apparent diminution of the rate of transition. For this purpose the liquid was placed in a small tube kept in position by a plug of glass-wool, so that the liquid never came in contact with the mercury. If, however, the sealed tube containing liquid iron carbonyl, or a solution of it in ether, be heated to any temperature between 60° and 100° C., while exposed to light, no solid separates even after several hours, whereas below 50° C. solid is formed in about half an hour. This is also true when the liquid is under a pressure of 50 to 100 atmospheres of carbon monoxide. The solution which had been exposed to light at a temperature above 60° C. even on cooling gave no deposit of solid, showing that no decomposition had been caused by the action of light at these temperatures. And this is confirmed by the fact, which will be proved later, that if solid had been produced in solution it would at this temperature have formed a solution of an intense green colour.

The decomposition of liquid iron carbonyl dissolved in ether, amylene, or petroleum-ether (B.P., 30° to 40° C.) by light, takes place slowly at the temperature of liquid

air. The solutions become solid, and, after exposure to sunlight for about three hours inside a vacuum vessel of liquid air, and then allowing to warm up in the dark, a faint deposit of solid was observed.

In spite of the fact that pressure is not effective in preventing the decomposition by light, the reaction is reversed slowly under a slight pressure of carbon monoxide at the ordinary temperature in the dark. Tubes containing iron carbonyl alone, or in solution, which had been exposed to light so that they contained some of the solid carbonyl, on standing at the ordinary temperature in the dark for some weeks, were found to contain no solid; so that the solid had absorbed the carbon monoxide which had been evolved, and had been completely re-converted into the liquid. These observations are of considerable interest and importance in their bearing on the question as to whether the action of light is exothermic or endothermic. If the light reaction is not endothermic, then we must assume the action of carbon monoxide on the solid carbonyl, and the change backwards into the liquid carbonyl is attended by an absorption of heat. Further experiments must be made to settle the question.

When solid iron carbonyl is heated alone, as stated above, no change takes place below 100° C.; at this temperature a solid and a green liquid are formed. But if the solid be heated with liquids such as ether, petroleum-ether, or toluene, change begins at about 50° C., the solid decomposes, and the liquid acquires an intense green colour. The intensity of the green colour is so great that the solutions are almost opaque, even in thin layers; in more dilute solutions the absorption spectrum showed a distinct band in the yellow. These green solutions, on exposure to light, again deposit yellow crystals of the solid carbonyl, and become colourless.

In order to determine what kind of light was most effective in inducing this decomposition, small tubes containing a 10 per cent solution of iron pentacarbonyl in ether were exposed in different parts of a solar spectrum and also to sunlight under different coloured screens. It was found that most rapid decomposition occurred in the blue, then green, closely followed by yellow, and lastly red: exposure under red glass produces roughly about one-tenth the amount of solid produced under blue glass in the same time.

Exposure of the liquid in quartz tubes to the electric arc causes slow decomposition only, and the acetylene flame is still less active.

Colouring the ether solution with dyes was also tried; cyanine and chlorophyll all *vw* rapid decomposition, azobenzene retards the decomposition slightly, isatin and alizarine somewhat more.

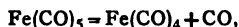
It has already been stated above that the decomposition occurs readily in solution in petroleum-ether and ether; the same is true of alcohol; in pure benzene the decomposition appears to be slower, but if the benzene contain traces of thiophene a black solid is deposited. Solutions of iron carbonyl in pyridine become dark red in colour when exposed to light, gas is evolved, but no solid is deposited except from strong solutions of about 50 per cent by volume. This is to be accounted for by the solubility of the solid in pyridine. Carbon bisulphide and nitrobenzene, which do not react with iron carbonyl in the dark, when exposed to light react with the formation of solid precipitates.

The most striking result observed was with solutions of iron carbonyl in nickel carbonyl. These solutions are of a much paler yellow colour than solutions of equal concentration in other solvents. Thus, for example, a 10 per cent solution of iron carbonyl in ether has just the same intensity of colour as a 30 per cent solution in nickel carbonyl. A 10 per cent solution deposits no solid after exposure to bright sunlight for several weeks, a 25 per cent solution (by volume) deposits no solid from the liquid, but solid is deposited in the vapour space above the liquid; a 50 per cent solution deposits some solid both in the liquid and in the vapour space.

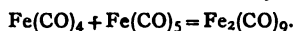
That no decomposition occurs in dilute solutions of iron carbonyl in nickel carbonyl is shown by the facts that no gas is evolved from these solutions, and that solid iron carbonyl is only sparingly soluble in nickel carbonyl. The absence of any change is not to be accounted for by the absorption of the active light by the nickel carbonyl, since the iron carbonyl has been shown above to be sensitive to light in the visible part of the spectrum which is not absorbed by nickel carbonyl. This fact was further confirmed by exposing a small tube full of iron carbonyl immersed in liquid nickel carbonyl to light, when decomposition was found to take place rapidly.

The following may be suggested as a simple hypothesis to account for the unique behaviour of nickel carbonyl as a solvent of iron carbonyl:—

The initial action of light on iron carbonyl might be represented by the equation—



the hypothetical iron tetracarbonyl produced may be assumed to combine at once with a molecule of iron pentacarbonyl to produce the solid nonacarbonyl, thus:—



If this molecular mechanism of the light reaction be admitted, then there is no reason why iron pentacarbonyl may not form an analogous body of feeble stability by combining directly with nickel tetracarbonyl, thus:—



A compound of this kind, though unstable in itself (since the vapour above the solutions contains iron carbonyl and the concentrated solutions deposit some solid), may yet be unacted upon by light. The existence of this compound is rendered probable by the fact that solutions of iron carbonyl in nickel carbonyl have such a pale colour compared to solutions of the same concentration in other solvents.

The observations on the action of light on iron carbonyl under different pressures of carbon monoxide, at different temperatures and in solution in various solvents, will be continued as soon as the necessary sunlight is available.

DISTILLED WATER SUPPLY FOR "WORKS" LABORATORIES.

By RICHARD SELIGMAN.

IN a "works" laboratory an adequate supply of distilled water is always a matter of importance, and in those situations where cheap gas is not available, its provision is often attended with considerable difficulty. When works are provided with steam it is a simple matter to obtain distilled water by running a pipe into the laboratory, and passing the steam through a condensing coil, but this method is not available where condensing engines are in use, or where the feed water is charged with volatile matter, for the condensed water will contain oil or other impurities.

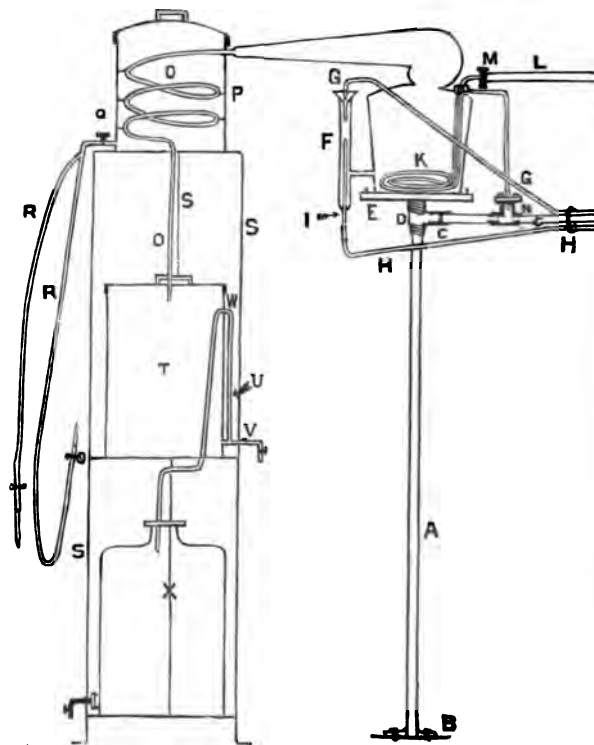
If a large number of analyses are made every day it generally happens that the glass wash-bottle does not meet the requirements of the worker. Not only has it to be frequently replenished and heated on a separate stove, but it is liable to breakage, and the process of washing, say, twenty or thirty precipitates with it is exceedingly laborious and slow. It is found convenient to supplant the wash-bottle by an elevated tank from which passes a suitable number of rubber tubes long enough to reach all parts of the bench. To the ends of the tubes are attached suitable glass nozzles. Each tube is closed by a pinch-cock on compressing which a stream of water issues from the nozzle, and may be used to expeditiously wash a large number of precipitates. The water in the tank is kept at the required temperature by a steam coil or by a stove.

For the complete installation the pieces of apparatus necessary are:—

1. Still.
2. Condenser.
3. Receptacle for condensed water.
4. Elevated tank supplied with a heating coil.

The following arrangement, which has given satisfactory results in daily use, and which can be cheaply and easily installed, has the great advantage that it can be made from apparatus with which most works laboratories are already provided.

Where the working bench is in the middle of the laboratory, so that both sides are utilised, economy of space is desirable. The still is therefore supported on an upright of $\frac{3}{4}$ -inch iron (A) 3 feet 6 inches long, firmly screwed to the



bench-top by means of a flange (B). This structure is steadied by the waste-steam pipe (C), which crosses from the wall at a convenient height above the heads of the operators. On the top of A is a T-joint (D) into which the plugged end of C is screwed, and which also carries the platform (E), on which rests the still. The still has a capacity of five litres, and has a constant level supply (F), to which the water is brought by a $\frac{3}{4}$ -inch compo-pipe (G) carried along the steam pipe C. The overflow is carried off by the pipe (H) likewise suspended from C. The pressure in the still is regulated by adjusting the height of the pipe (I), which at the same time acts as a safety valve. Heat is supplied by a flat coil (K) of $\frac{3}{4}$ -inch copper pipe, tinned outside, of which three turns are found to suffice. The coil hangs $\frac{1}{2}$ -inch above the bottom of the still. Live steam from the boilers is supplied by the pipe (L), joined by the coupling (M) to the coil K. The waste steam passes by the coupling (N) to a branch on the pipe C, which in its turn is closed by a steam trap of any suitable form. The neck of the still is soldered into the mouth of the block-tin condensing worm (O), which makes three turns inside the copper tank (P). This latter is 8 $\frac{1}{2}$ inches in diameter and

9½ inches high, is well tinned inside, and serves the double purpose of condenser and supply tank.

It is filled once a day with distilled water, and the steam passing through o, whilst being condensed itself, heats the water surrounding it, and maintains it at a suitable temperature for use. Close to the bottom of p is the tap (q), to the neck of which the rubber tubes (r r) carrying pinch-cocks and glass nozzles are attached. The tank p stands on the tripod (s), 4 feet above the bench. A second stage in this tripod carries the tinned copper tank (τ) (8½ inches in diameter by 14 inches high), into which the hot water delivered by the worm, o, falls. Close to the bottom of τ is a T-shaped pipe (v). The upright arm carries a glass tube (u), the horizontal arm a tap for drawing off the cold water in the bottom of τ. u serves the double purpose of gauge and overflow, being bent back on itself at w, and delivering into the bottle (x). In order to prevent syphonage, a minute hole is blown in u at the top of the bend w. Thus only cold water flows into x.

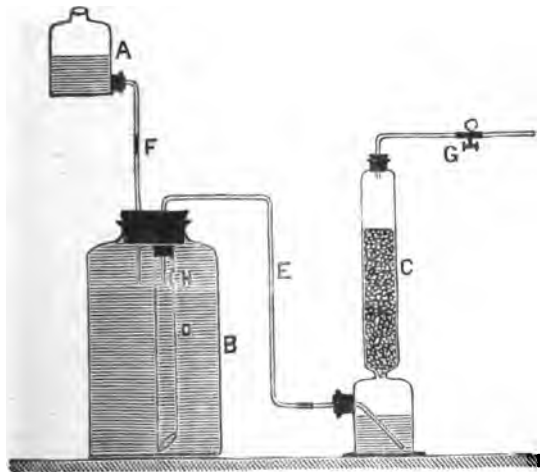
The apparatus described works automatically, except that each day the bottle x has to be removed, and its contents emptied into the tank p. It gives a supply ample for about 200 analyses weekly, as well as for those other purposes requiring the use of distilled water.

A NEW FORM OF GAS GENERATING APPARATUS.

By A. W. GREGORY.

THE apparatus here described has been found very efficient for large and continuous supplies of sulphuretted hydrogen.

A is a tubulated bottle placed in an elevated position, F a delivery tube leading from A to the reservoir bottle B, which is simply a wide-mouth bottle of about five litre capacity.



D is a wide glass tube connected with E by means of a cork and has an opening at H, just above the termination of E.

C is a "tower," the upper part of which is filled with iron sulphide.

A and B are filled with dilute hydrochloric acid and the clip G opened.

Acid flows into C, and sulphuretted hydrogen is generated.

When the clip is closed, the partially spent and denser acid returns to B, and is conducted to the bottom of the reservoir by means of the tube D.

On again opening the clip, fresh acid goes to the tower through the opening H.

Each time the clip is opened the acid of lowest specific gravity and, consequently, the strongest acid in the reservoir, is brought into contact with the iron sulphide.

THE CHEMICAL SEPARATION OF THE RADIO-ACTIVE TYPES OF MATTER IN THORIUM COMPOUNDS.*

By HERMAN SCHLUNDT and RICHARD B. MOORE.

Continued from p. 17).

Curves of Recovery and Decay (continued).

THE far greater increase in activity exhibited by the residues obtained in the pyridine and fumaric acid separations—the residue from the latter showing an increase of more than 50 per cent over the initial value—demands further explanation. Moreover, the fact that the maximum values are reached in about one-fourth the time required for the ammonia residue to come to a maximum, indicates that the distribution of the several types of matter in the separation with fumaric acid and pyridine differs in some respects from the separation by means of ammonia. It will be shown that this difference arises from the matter causing imparted activity—the fumaric acid and pyridine effecting a separation of its components. From his experiments on the matter causing imparted activity of thorium (*Phil. Mag.*, 1903 [6], v., 95), together with the experiments carried out by Rutherford and Miss Brooks ("Radio-activity," p. 260), Rutherford concluded that the imparted activity was complex. On the basis of the results obtained by exposing a negatively-charged wire for short intervals to the thorium emanation, Rutherford pointed out that they could be satisfactorily explained by assuming the presence of two products—one not radio-active, that is, emitting no rays, with a rapid rate of change, the other resulting from it, radio active, but with a slower rate of change. In the first change, one-half of the matter disintegrates in fifty-five minutes, in the second one-half changes in eleven hours. Quite recently, however, Rutherford (*Phil. Trans.*, A., 376) has stated that the results obtained might be explained equally well by assuming that the matter with the slow rate of change and giving off no rays is the first product, and that this inactive matter produces a radio-active product with the rapid period of decay. About the same time Miss Slater (*Phil. Mag.*, May, 1905) succeeded in effecting a partial separation of the two types of matter constituting the imparted activity, which had been deposited on a negatively-charged wire by a long exposure to the emanation from thorium. She did this by the action of the cathode rays and also by heating to a high temperature. The results of her experiments, Miss Slater states, can be satisfactorily explained on the theory, independently advanced by her, that the first product is inactive and has the slow period of decay, and that it changes into a radio-active product with a rapid period of decay. Miss Slater designated the first product, which emits no rays and has the slow rate of change, ThA, and the second product which emits radiations and has the rapid rate of decay, ThB. We shall use this terminology in connection with the further discussion of the initial peculiarities of the curves of recovery and decay. The view advanced by Rutherford and Miss Slater also affords a satisfactory explanation for the initial irregularities in the curves of recovery and decay of the precipitates and residues obtained in the separations with fumaric acid and pyridine.

When thorium is precipitated with fumaric acid, the precipitate, instead of containing both ThA and ThB, as is the case when the precipitation is made with ammonia, contains only ThB. The first product of the imparted activity, ThA, is soluble in excess of this reagent, and

* From the *Journal of Physical Chemistry*, ix., No. 8, p. 682.

hence is found in the filtrate with ThX. As ThA undergoes change without giving off rays, the initial activity of the filtrate has the same value as that obtained with ammonia. The precipitate, which contains thorium and ThB, is not in radio-active equilibrium, but before the thorium has formed even a few per cent of ThX, ThB has changed practically completely into a final inactive product, one-half of the change taking place in fifty-five minutes. Hence, as ThB is responsible for about one-half the ionisation produced by the precipitate initially, its rapid decay accounts for the rapid decrease in activity of the precipitate during the first four hours after precipitation. It will be noted that by deducting the effect due to the non-separable activity from the curve representing the combined effect of the non-separable thorium activity and ThB (Curve *c*, Plate II.), the difference curve drops to half value in about one hour, which is the characteristic rate of decay of ThB.

From the above it can readily be seen that if the processes of precipitation and filtration are carried out slowly, so that the first readings are made four or five hours after the first precipitation, the curves will show no initial drop, and the result will be the same as that obtained by Rutherford and Soddy with twenty-three precipitations by means of ammonia.

In the residue from the filtrate ThX and ThA are in equilibrium at the outset, but the residue as a whole is not in equilibrium, owing to the absence of the radio-active product ThB. This product, however, is produced from ThA at a much faster rate than ThX decays, and since ThB changes at a still faster rate, the activity of the residue shows a rapid initial rise, reaching a maximum about five hours after separation.

The residue obtained in the ammonia separation being free from both ThA and ThB shows a smaller rise initially, because the ThA produced by ThX is very small in proportion to that present in the fumaric acid residue, and since the increase in activity of the residue depends upon the amount of ThB produced from ThA, it follows on this view that the initial increase in activity should be considerably greater when the residue contains the normal amount of ThA, as is the case in the fumaric acid separation.

The initial peculiarities of the curves representing the decay and recovery of the products obtained in the pyridine separation are not quite as marked as in the fumaric acid separation, probably because the separation of ThA and ThB was not quite complete.

The correctness of the view here presented was tested by several experiments. In connection with the fumaric acid separation in the early part of this paper, two methods were given by which ThA was separated from the other products. In one of these experiments, ThA was removed from the *precipitated thorium*, obtained with ammonia, by means of fumaric acid, and in the other one, ThA was removed from the *filtrate* obtained in the fumaric acid precipitation by the addition of ammonia. The curve marked ThA $\rightarrow$ ThB, Plate I., is the theoretical curve of the residue ThA, while the plotted points represent the experimental values obtained. It will be observed that this curve closely resembles the initial portion of the curve of the fumaric acid residue (C, Plate II.).

In another experiment the thorium was precipitated once with fumaric acid and filtered off; the precipitate was then quickly re-dissolved in dilute nitric acid, and after the addition of a trace of lead nitrate, the solution was at once subjected to electrolysis for five minutes, a rotating cathode being employed with a current density of about six amperes per 100 sq. c.m. of cathode surface. The electrodes were washed and dried quickly and then tested for activity. The slight deposit of lead peroxide on the anode was found to be practically inactive and subsequently showed no change, but the slight deposit on the cathode was highly radio-active, its activity decreasing according to an exponential law, decaying to half value in fifty-five minutes, and finally becoming entirely inactive

and remaining so. This experiment shows that the precipitate of thorium obtained with fumaric acid contains the product ThB, and not ThA: for, according to the experiments of Pegram (*Phys. Rev.*, 1903, xviii., 424) on the electrolysis of thorium nitrate solutions, ThA is deposited on the anode under the conditions of our experiment, but the anode deposit in our experiment was inactive, and showed no change with the time, as it should have done if ThA had been deposited. Our thorium nitrate solutions, when subjected to electrolysis, behaved in the manner described by Pegram—the anode deposit being quite active, the activity decaying to half value in eleven hours after showing the initial rise characteristic of ThA, while the cathode deposit had a slightly slower rate of decay than the ThB similarly obtained from the thorium nitrate solution from which ThA and ThX had been removed.

(To be continued).

CITY AND GUILDS OF LONDON INSTITUTE.

FINSBURY TECHNICAL COLLEGE PAST STUDENTS' DINNER.

ON Saturday last, the 13th inst., Prof. MELDOLA, F.R.S., took the chair at the first Annual Dinner of the Past Chemical Students of the Technical College, Finsbury, at the Hôtel Previtali.

In addition to the Lecturers and Demonstrators of the Chemical Department, 70 Past Students gathered together, including Dr. M. O. Forster, F.R.S., Dr. G. T. Moody, Dr. O. Silberrard, Dr. C. H. Desch, Messrs. A. R. Ling, J. L. Baker, F. G. Collier, T. J. Underhill, A. Greeves, F. H. Carr, and many others. The Principal of the College, Prof. Silvanus P. Thompson, F.R.S., was prevented by ill-health from being present, and Prof. W. J. Pope, F.R.S., was among the past students who were unable to come.

Prof. MELDOLA referred with pride to the number of past students, who had won distinction in the chemical world, who were gathered around him; Finsbury was one of the earliest Technical Colleges, and had a record of a quarter of a century's usefulness to the technical industries of the country.

Dr. Moody, who proposed "The College," said that this year was a most appropriate one for the first Annual Dinner, as their head, Prof. Meldola, now held the highest distinction the Chemical Society had to offer, the office of President.

A well-chosen musical programme added to the success of the evening.

OBITUARY.

DR. HERMANN JOHANN PHILIPP SPRENGEL, PH.D., F.R.S.

WE regret to announce the death of Dr. Hermann Sprengel, which occurred suddenly on Friday last, the 12th inst., at the age of 72.

Dr. Sprengel was born in the year 1834 at Schillerslage, near Hanover, in Germany, and was educated at the Universities of Göttingen and Heidelberg, where he took his degree in 1858. Coming to England in the following year he worked first at Oxford, and then at the College of Chemistry, Guy's and Bartholemew's Hospitals. The most important of his researches were concerned with the two extremes of the gaseous state of matter, viz., high vacua and detonating agents, but it is by the well-known Sprengel mercury pump that his name will be best remembered, and were it not for this discovery it is highly probable that electric lighting would not be in such an advanced stage as it now is, to say nothing about other researches on radiant matter, X-rays, electrons, &c., which have depended to such a great extent on the perfection of the apparatus used for obtaining high vacua.

NOTICES OF BOOKS.

Elements of Quantitative Analysis. By G. H. BAILEY, D.Sc. (Lond.), Ph.D. (Heidelberg). London: Macmillan and Co., Ltd. New York: The Macmillan Company. 1905.

THERE is much in this little work to commend it both to teachers and students, although in some respects it is not above reproach. To illustrate the former statement, first it is only necessary to call attention to Part II. on Volumetric Analysis, in which well-chosen methods are most carefully described, and, indeed, accuracy of detail is a marked characteristic of the book. Instructive examples are given of the examination of minerals containing the rarer elements, and the description of, for instance, the extractions of uranium from pitchblende and of the rare earths from cerite will be found useful by advanced students. Part I. treats of the general operations with which the beginner should make himself familiar, and the author's attention to detail and minuteness of description should both tend to assist especially those who are working with but little or no assistance from a teacher. Parts III. and IV. are devoted to the Analysis of Minerals, many of which are studied, and to Technical Products, a few typical analyses being outlined, such as those of fertilising agents, soap, &c. The chief defect of the book is the comparative neglect of electrolytic methods, with the exception of the estimation of copper and lead, which are very briefly described, although it is now generally recognised that electrolysis provides very often the most accurate and rapid methods of determining some metals, and every student should be given practice in the manipulation of electrolytic apparatus.

Glue, Gelatine, and their Allied Products. By THOMAS LAMBERT. London: Charles Griffin and Co., Ltd. 1905.

THE rapid growth of the glue and gelatine industry makes the appearance of a text-book devoted exclusively to its consideration particularly welcome, and although this work is not very exhaustive, it gives a sufficient account of modern methods of working, chiefly from a practical standpoint, the preparation of glue and gelatine being described fully, while a very valuable collection of recipes for the manufacture of liquid glues, cements, &c., is included. These recipes have been collected from periodical literature, and are believed by the author to be trustworthy. The chapter on the residual products from glue and gelatine gives the manurial values, and composition of fish and bone residues, while the last chapter deals with the analysis of the raw and finished products. This part is in some respects the least satisfactory in a book which is otherwise of real value. For instance, the details of the determination of fat in a Soxhlet's extractor are inadequate, the temperature at which the operation is conducted not being stated, while the number of times of syphoning recommended would probably be found by experience to be insufficient for the complete extraction. It appears ill-advised to include the analysis of manures, unless more space can be devoted to a description of the processes, and the greatest stress must be laid upon the exceedingly unsatisfactory directions given for the preparation of the standard solutions used in volumetric work. As an example, it may be mentioned that to prepare normal sodium hydrate it is suggested that 40 grms. of pure or 42 grms. of ordinary caustic soda should be dissolved, and the solution made up to a litre without any preliminary purification in the latter case, or any standardisation with a more accurately prepared acid solution, while the fact that the value of each c.c. is given to three places of decimals would lead the inexperienced to suppose that a high degree of accuracy might be expected when such a solution was used.

The Book of the Rothamsted Experiments. By A. D. HALL, M.A. (Oxon.). London: John Murray. 1905.

IN this account of the work of Lawes and Gilbert an excellent summary is given of the Rothamsted Memoirs, and the author, writing more especially for agricultural teachers, has emphasised the chief results obtained, and dwelt upon the main purpose of the experiments—to ascertain how the plant grows and how it derives its nutriment from the soil. While the requirements of the teacher are specially kept in view, the author has written so clearly and lucidly that the agricultural student will find nothing in the book above his head, and it will serve to broaden and deepen his knowledge if he reads it in conjunction with the usual text-books. Moreover, the farmer will benefit by the perusal of an account of the most elaborate and complete set of field experiments ever conducted, although he may not find that thus he has actually added to his knowledge of how to manage his own land with the greatest success, for the experimental conditions were purposely often exceedingly unpractical, and the man who takes a strictly utilitarian view of all scientific work would be led to doubt the usefulness in practice of many of the experiments. But the student of deeper insight will recognise the value of this seemingly useless work, by which material has been steadily collected during the past sixty years for the expert to sift and base his opinions upon. The discussion of experiments on feeding is included, besides some miscellaneous researches on sewage irrigation, the composition of the wheat-grain and its mill products, &c., while, naturally, the bulk of the book is devoted to experiments on the growth of various crops, grown either continuously year after year upon the same land, or in rotation. Each chapter is admirably illustrated by curves and diagrams, and summaries are frequently given. The importance of the work of Lawes and Gilbert is being more fully recognised as time goes on, and this general account, by the able director of the station, of the results so far obtained must be acknowledged to be the most complete and interesting which has yet been issued.

Die Elektrolyse Geschmolzener Salze. Zweiter Teil. ("The Electrolysis of Fused Salts." Part II.). By RICHARD LORENZ. Halle-a-S.: Wilhelm Knapp. 1905.

THE second part of this monograph, the twenty-first volume of the series on Applied Electrochemistry, deals with Faraday's law, the migration of the ions, and conductivity. The author has brought together all that is known of these subjects, adding his own observations and explanations, so that the work is of more value than a mere compilation. The order adopted is that of systematic electrochemistry, and as no similar work exists dealing exclusively with the electrochemistry of the fused state, the monograph will be heartily welcomed, especially as it adds considerably to our knowledge of the theory of this branch, which has been, comparatively speaking, neglected. The author is of the belief that the time is not far distant when much quantitative work, performed from theoretical points of view, will be done in this region, and it will be universally recognised, when this time comes, that he has done much to bring about this end, both by his work of investigation, and secondarily by the preparation of this exhaustive monograph on the subject.

Les Quantités Élémentaires d'Électricité, Ions, Electrons, Corpuscules. ("The Elementary Quantities of Electricity, Ions, Electrons, Corpuscles"). Edited by HENRI ABRAHAM and PAUL LANGEVIN. Two Volumes. Paris: Gauthier-Villars. 1905.

THESE volumes belong to the second series of memoirs on physical subjects, issued by the French Physical Society. The papers in it are reproduced in the alphabetical order of the authors' names, the papers of each author being arranged chronologically. This arrangement simplifies reference to the books, and seems on the whole to be the

most convenient. The editors have carefully selected the most important and characteristic papers of all the authors, and the translators have frequently added explanatory notes. In the case of radio-activity the editors have confined themselves to those articles which deal with the emission of the α - and β -rays, and have altogether passed over the study of the transformations which occur in radio-active substances. Similarly in other branches, in order to keep within moderate bounds as regards size, they have been obliged to exercise their discretion in neglecting certain aspects, but this has been done with admirable judgment, and has in no way lessened the value of the book, which contains a collection of all the work that has been published on the nature and properties of ions, electrons, or corpuscles.

Handbuch der Anorganischen Chemie. ("Handbook of Inorganic Chemistry"). Edited by Dr. R. ABEGG. Vol. II., Part II. Leipzig: S. Hirzel. 1905.

In the second part of Vol. II. of this handbook the elements of the second group of the periodic system are treated. The theoretical aspects of the chemistry of these elements is emphasised, great stress being laid upon the results of investigations from a physico-chemical point of view. For each element a compendious bibliography is given, and special articles on the atomic weights have been prepared by Professor Brauner, the values having been very carefully re-calculated for the purpose. Spectral phenomena are only lightly touched upon, the full discussion of spectrum analysis being relegated to the ninth and general volume, and some inorganic compounds of only limited interest have been excluded, though the most important and characteristic organic salts, such as formates, acetates, &c., and the organo-metallic bodies, are regarded as coming within the sphere of the book. The need of an authoritative text-book of pure chemistry of the nature of this treatise has undoubtedly been felt, and this need seems likely to be admirably supplied by Dr. Abegg's book.

La Théorie Moderne des Phénomènes Physiques Radio-activité, Ions, Electrons. ("Modern Theory of Physical Phenomena, Radio-activity, Ions, Electrons"). By AUGUSTO RIGHI. Translated by EUGENE NÉCULCÉA. Paris: L'Eclairage Electrique. 1906.

THE French translation of Prof. Righi's interesting book has been prepared from the second Italian edition. An English version of the same book was noticed in these columns a short time ago, and little remains to be added to the comments then expressed. The translator has provided some explanatory notes, especially on the subject of radio-activity, giving fuller details of the phenomenon of scintillation, of induced radio-activity, and of the heat evolved by a radium salt, &c., and Prof. Lippmann has written a short introduction to the book, in which we notice a curious mistranslation in a quotation from the original relating to the supposed elastic deformations of the ether; otherwise the translation has been accurately and carefully performed, and the book should appeal to a large circle of readers in France.

Physikalische Krystallographie. ("Physical Crystallography"). By P. GRÖTH. Fourth Edition. Leipzig: Wilhelm Engelmann. 1905.

ALTHOUGH the author of this work speaks of it in the preface as an elementary text-book of crystallography, the fourth edition can lay claim to being one of the most complete treatises on the subject which has yet been written, containing accounts of all the most recent researches. The book is now divided into three sections; the first enumerates and discusses the properties of crystals in general, while in the second a systematic description of the various classes of crystals is given with numerous examples. The last section deals with methods of investigating crystals, th

calculation and graphic representation of crystal forms, the measurement of angles and refractive indices, and their optical examination in polarised light. In the short section on the apparatus for the determination of hardness and elasticity of crystalline substances, only the simplest instruments are described and those most suited for the use of beginners. The book is fully illustrated throughout, and the author has overlooked no important advance in our knowledge of the science, while his compendious work throws considerable light upon the question of the correlation of composition and crystalline forms.

Traité de la Fabrication de la Soude d'Après le Procédé à l'Ammoniaque. ("Treatise on the Manufacture of Soda by the Ammonia Process"). By H. SCHREIB. Translated from the German by Dr. L. GAUTIER. Paris and Liège: Ch. Béranger. 1906.

THE author of this work has had great experience of the working of the ammonia process for the manufacture of soda, and is well qualified to report upon it, and in the absence of a book devoted exclusively to the consideration of the method, the translation of it will undoubtedly be welcomed by French-speaking chemists and manufacturers. The book describes fully the various modifications of the process, appraising the practical value of each, and gives many plans and illustrations, so that the increasing number of those who are giving up the practice of the Leblanc method and are interested in the installation of new factories will find it a valuable aid in their work, while the chemists and managers of ammonia-soda factories may be strongly advised to add it to their libraries.

CORRESPONDENCE

ACTION OF LIGHT ON GLASS.

To the Editor of the Chemical News.

SIR,—In the course of an article which appeared in the CHEMICAL NEWS (vol. xci., p. 192) on the "Action of Ultra-violet Light upon Glass containing Manganese," I notice a remark upon the violet colouration produced upon such glass by the action of sunlight at the high altitude of Uyuni in Bolivia.

It may be of interest to note that in this district (Northern Chile) the same effect readily takes place at the sea-level. A fragment of common table-glass shows a decided violet tinge after less than a month of exposure to the sun. In the various specimens which I have examined, manganese has been present.—I am, &c.,

OSWALD H. EVANS, F.G.S.

Taltal, Chile.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances de l'Académie des Sciences. Vol. cxli., No. 25, December 18, 1905.

At the annual Meeting of the Academy of Sciences, reports of the work of the various prizewinners were read and adopted. Among others, prizes were awarded as follows:—

Prix Jecker to MM. Sabatier and Senderens—"On the Catalytic Effects of a certain number of Metals."

Prix Cahours to MM. Binet du Jassoneix and Kling.

Prix La Caze to Albert Colson for his researches in organic, mineral, physical, and industrial chemistry.

Berichte der Deutschen Chemischen Gesellschaft.
Vol. xxxviii., No. 16, 1905.

Quantitative Determination of Bismuth, and its Separation from Copper, Cadmium, Mercury, and Silver.—Arthur Staehler and Wilhelm Scharfenberg.—Bismuth cannot be determined as Bi_2O_3 in presence of hydrochloric or sulphuric acid, as basic salt is precipitated. The determination as sulphide gives results which are always too high, because the sulphide retains sulphur, and is, moreover, very easily oxidised. Better results are obtained by Rose's method of fusing the sulphide with potassium cyanide, and thus converting into metal. The precipitation of the phosphate or arsenate of bismuth seems to be the most generally applicable method, and the most satisfactory. The authors have found that the biphosphate of bismuth is quite insoluble in phosphoric acid solution, and they can thus separate bismuth from many other metals, e.g., copper, cadmium, silver, and mercury, whose phosphates are soluble in phosphoric acid. Bismuth can be separated quantitatively by means of sodium phosphate in acid solution, without the reaction being affected by the presence of chlorides or by the strength of the nitric acid used. A 10 per cent solution of trisodium phosphate was used, and a bismuth solution prepared by dissolving pure bismuth in strong nitric acid. This bismuth solution was diluted with water and boiled, any precipitate of basic salt being removed by the addition of a little nitric acid. The boiling sodium phosphate solution was then added drop by drop to it, stirring gently all the time. The whole was boiled for a short time, and then the precipitate settled readily. It was filtered off hot, and washed with 1 per cent nitric acid containing a trace of ammonium nitrate, the filtrate always being tested for bismuth. The precipitate was dried at 120° in a Gooch's crucible, ignited over a Bunsen burner for five to ten minutes, and weighed as BiPO_4 . To separate bismuth and cadmium, the bismuth is first precipitated as above, and then, in absence of hydrochloric acid, the cadmium is separated electrolytically from ammoniacal solution to which some potassium cyanide has been added. In presence of hydrochloric acid the cadmium is separated as CdS , by passing sulphuretted hydrogen into the filtrate from the bismuth. The sulphide is then dissolved in nitric acid and electrolysed, after the addition of caustic potash and potassium cyanide. Mercury is quickly and accurately separated from hot ammoniacal solution as sulphide, quite free from excess of sulphur, after the bismuth has been precipitated as phosphate. Lead phosphate is separated quantitatively from neutral solution by means of sodium phosphate. However, the microscopic examination of the precipitate shows that it consists of at least three different phosphates, which by prolonged boiling are converted into tertiary lead phosphate. This behaviour indicates the way to separate lead and bismuth in presence of hydrochloric acid. Both phosphates are first precipitated with sodium phosphate, and the lead is then removed by boiling with 1 per cent nitric acid. A second method, which would undoubtedly be successful, is to heat to 500° a mixture of bismuth oxychloride and lead chloride in a current of sulphur monochloride and chlorine. At 447° the bismuth goes over as chloride, while the lead chloride remains behind.

Palladium.—A. Guthrie and A. Krell.—Alkylated anilines and their haloid hydrates exhibit a characteristic behaviour towards palladous haloids. When a small quantity of chlor- or brom-hydrate of the base employed acts upon excess of palladous chloride or bromide, double salts result, and on reversing the reaction the palladosamine derivatives are obtained, which is not the case if the aromatic amines are alkylated in the side chain. In this case the tendency to form double salts is greatly increased, and crystalline products are obtained, both from dilute alcohol and from the corresponding dilute haloid acid, in whatever order the reagents are added, and also when the free base or an alcoholic solution of it acts upon the palladous haloid. The corresponding palladosamine de-

rivatives are obtained:—(1) By the action of an alcoholic solution of the corresponding base upon neutral aqueous solutions of chloro- or bromo-palladites; (2) by heating aqueous solutions of the double salts freed from excess of acid; (3) by allowing small quantities of the palladous haloid solution to act upon an excess of the base dissolved in alcohol. These derivatives of palladosamine are characterised by their light colour and by the fact that they are difficultly soluble, although they crystallise very well from large quantities of dilute alcohol. They dissolve easily in warm concentrated ammonia, and are then converted into palladosamine chloride or bromide, ammonia taking the place of the organic base.

Action of Bromine upon Trimethylamine.—James F. Norris.—By the action of bromine on trimethylamine a compound is obtained to which the author ascribes the formula $(\text{CH}_3)_3\text{NHBr}\cdot\text{Br}$. To confirm this formula the author brings forward the following facts:—(1). The substance is formed by the action of bromine upon trimethylamine bromhydrate, $(\text{CH}_3)_3\text{N}\cdot\text{HBr} + \text{Br} = (\text{CH}_3)_3\text{NHBr}\cdot\text{Br}$; (2) as it seemed possible that this reaction might proceed as follows: $(\text{CH}_3)_3\text{NHBr} + \text{Br}_2 = (\text{CH}_3)_3\text{NBr}_2 + \text{HBr}$, and lead to the formation of a bromine addition product, a careful investigation was performed to ascertain whether hydrobromic acid was split off, but the result was negative; (3) hence it was to be concluded that the hydrobromic acid necessary for the formation of $(\text{CH}_3)_3\text{NHBr}\cdot\text{Br}$ from trimethylamine and bromine resulted in a secondary reaction in which a part of the amine was decomposed by the halogen, hydrobromic acid being formed; (4) analysis showed that the total amount of bromine in the substance corresponded to the formula $(\text{CH}_3)_3\text{NHBr}\cdot\text{Br}$. On addition of water and potassium iodide exactly half the bromine present was set free.

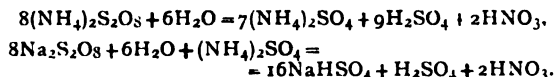
Determination of Nitrous Acid.—F. Raschig.—The author has investigated the determination of nitrous acid, using the method described by Meisenheimer and Heim (*Berichte*, 1905, xxxviii., 3834; *CHEMICAL NEWS*, 1905, xcii., 304); however, he does not measure the nitric oxide, but prefers to determine the iodine separated by titration with thiosulphate, which is naturally much quicker than measuring the gas. This method is useful only when the far more convenient permanganate oxidation method cannot be employed. Lunge added the nitrite solution very slowly to a known quantity of acid, $\frac{1}{2}\text{N}$. permanganate solution, warmed to 40°C . This method has the disadvantage that the errors in the determination are the greater the stronger the nitrite is, and, moreover, the decoloration of the permanganate proceeds so slowly towards the end that it is very easy to add too much nitrite, and this error cannot be rectified by a back titration. Moreover, sometimes manganese peroxide separates and cannot be removed by fresh additions of nitrous solution. All these drawbacks are avoided by the author's method. He uses an excess of about 20 per cent of permanganate. If necessary acid is added (this is of course superfluous in the case of nitroso acids), and after two minutes all nitrous acid is oxidised to nitric. Then the excess of permanganate is determined, the best process being that of Volhard, in which potassium iodide is added, and the iodine set free is titrated with thiosulphate.

Quantitative Determination of Bismuth and its Separation from the Heavy Metals as Phosphoric Acid Salt.—H. Salkowski.—The author recommends the precipitation of bismuth as phosphate, if the precipitation liquid contains no hydrochloric acid or chloride; the only free acid which may be present is nitric acid. He has obtained satisfactory results in separating bismuth from copper and also from lead. The almost complete insolubility of bismuth phosphate in dilute nitric acid provides a convenient test for showing the presence of bismuth qualitatively.

Oxidations with Silver Peroxide. I. Oxidation of Oxalic Acid.—R. Kempf.—A very large number of

organic substances at the ordinary temperature immediately take away the brown colour of a mixture of a solution of potassium persulphate with a silver nitrate solution, an oxidation process taking place. As the silver peroxide is regenerated, so long as persulphate is present a very small quantity of the silver salt suffices to cause all the persulphate to react. If a solution of oxalic acid is added to a solution of sodium or ammonium persulphate in dilute sulphuric acid no reaction occurs, but as soon as small quantities of a silver salt are added a violent reaction takes place, accompanied by spontaneous warming and frothing. The oxalic acid is quantitatively oxidised to carbon dioxide. This process may be used to estimate volumetrically the active oxygen in persulphates, an excess of oxalic acid being used and titrated back with potassium permanganate solution, and the process is found to be more reliable and accurate than any other.

Oxidations with Silver Peroxide. II. Formation of Nitric Acid from Ammonium Sulphate.—R. Kempf. —When silver sulphate is added to a solution of ammonium persulphate in dilute sulphuric acid, the brown colour which first appears always vanishes after some days, although no oxygen is evolved, nor is any persulphuric acid still present in the liquid. Considerable quantities of nitric acid are to be found in the liquid, and its formation provides an explanation of the phenomena observed. The nitric acid can only be formed by the oxidation of the ammonium salt at the ordinary temperature in acid solution by means of silver peroxide. The author has found that sodium persulphate similarly converts zinc ammonium sulphate into nitric acid, the yield being almost quantitative in all cases, so that neither nitric acid nor free nitrogen can result to an appreciable extent. The reaction occurs according to the following equations:—



The velocity of the reaction is very small. In absence of a silver salt a persulphate solution cannot oxidise combined ammonia in sulphuric acid solution to nitric acid, and thus the energetic oxidation action is due only to the silver peroxide and not to the persulphuric acid or its products—ozone (which is formed in large quantities when a solution of persulphate in sulphuric acid is warmed), Caro's acid, or hydrogen peroxide. In the experiments above described the ammonium salt was present in great excess; if excess of persulphate is used, only about 69 per cent. of the ammonium present is converted into nitric acid. The general results of the author's work on the oxidation of ammonium salts to nitric acid may be summarised as follows:—1. Alkali persulphate oxidises ammonium salts in sulphuric acid solution in presence of silver sulphate almost quantitatively to nitric acid at the ordinary temperature. 2. In the absence of silver salts this oxidation does not take place. 3. In alkaline solution alkali persulphate oxidises free ammonia to nitric acid at the ordinary temperature without any catalyser. 4. (According to Marshall), in ammoniacal solution alkali persulphate oxidises ammonia in presence of silver sulphate only to elementary nitrogen.

Vanadium Sesquisulphates.—Arthur Stähler and Heinz Wirthwein. —Using the same method as that adopted for the preparation of titanium sulphuric acid (*Berichte*, 1905, xxxviii., 2620), the authors have prepared a complex vanadium sesquisulphate sulphuric acid of the composition $\text{V}_2(\text{SO}_4)_3 \cdot \text{H}_2\text{SO}_4 + 12\text{H}_2\text{O}$. From this acid two coloured salts could be prepared, viz., the ammonium and rubidium salts. When gently heated the acid gives up water and sulphuric acid, and yields yellow anhydrous vanadium sesquisulphate. This, like the corresponding sesquisulphates of iron, chromium, and titanium, is insoluble in water and sulphuric acid. When the vanadium sesquisulphate sulphuric acid is heated to a higher temperature, vanadium pentoxide is formed.

MISCELLANEOUS.

The Iron and Steel Institute.—*The Andrew Carnegie Research Scholarship.*—A Research Scholarship or Scholarships, of such value as may appear expedient to the Council of the Iron and Steel Institute from time to time, founded by Mr. Andrew Carnegie (Past-President), who has presented to the Iron and Steel Institute eighty-nine one-thousand dollar 5 per cent Debenture Bonds for the purpose, will be awarded annually, irrespective of sex or nationality, on the recommendation of the Council of the Institute. Candidates, who must be under thirty-five years of age, must apply on a special form before the end of February to the Secretary of the Institute. The object of this scheme of Scholarships is not to facilitate ordinary collegiate studies, but to enable students, who have passed through a college curriculum or have been trained in industrial establishments, to conduct researches in metallurgy of iron and steel and allied subjects, with the view of aiding its advance or its application to industry. There is no restriction as to the place of research which may be selected, whether university, technical school, or works, provided it be properly equipped for the prosecution of metallurgical investigations. The appointment to a Scholarship will be for one year, but the Council may at their discretion renew the Scholarship for a further period instead of proceeding to a new election. The results of the research shall be communicated to the Iron and Steel Institute in the form of a paper to be submitted to the Annual General Meeting of members, and if the Council consider the paper to be of sufficient merit, the Andrew Carnegie Gold Medal shall be awarded to its author. Should the paper in any year not be of sufficient merit, the Medal will not be awarded in that year.

Association of Teachers in Technical Institutes.—The following is a copy of a memorandum relating to the syllabus of instruction in practical organic chemistry which has been approved by the Council of the above Association, and forwarded to the Secretary of the Board of Education:—

Memorandum.—The Council of the Association of Teachers in Technical Institutions desire to draw the attention of the Board of Education to the following proposals regarding the syllabuses of certain science subjects, and respectfully request that the proposals shall be placed before the examiners and others concerned with these subjects with the view of modification in the directions indicated, and thereby it is believed materially improving the teaching of the said subjects.

Practical Organic Chemistry.—It is generally felt that the syllabus in its present form is most unsatisfactory, inasmuch as too great prominence is given to qualitative analysis, i.e., the detection in a mixture of two organic constituents from a given list. Whilst fully recognising that the student should be able to identify the more important organic compounds, we submit that the training necessary to enable him to analyse mixtures of the type set, is not such as really deepens his knowledge of the essential properties of the substance dealt with, since the majority of the tests and methods of separation applicable to such mixtures do not involve the fundamental characteristics of the compound nor the principal methods of organic chemistry. At present students have to curtail seriously the time which should be given to organic preparation, and devote their energies to the detection of the constituents of such mixtures as, for instance, potassium, cyanide, and cane-sugar, urea, and starch. We suggest that the object of the practical organic chemistry should be primarily the study of the reactions characteristic of the various classes of organic substances, emphasising as far as possible their applicability to a whole class, and not merely to individual compounds, with elementary analysis, the determination of certain physical constants and molecular weights in addition. The syllabus would, therefore,

read somewhat as follows:—Stage I (Aliphatic). Elementary qualitative analysis, determination of melting points and boiling points, reference of compounds to their classes, and preparation of derivatives; preparation and identification of a few typical compounds. Stage II. (Aromatic). Similar to Stage I. Stage III. Elementary quantitative analysis and determination of molecular weights. Preparation and detection of typical organic compounds.—I remain, on behalf of the Council, yours truly, J. WILSON, Hon. Secretary.

Two Iodo-mercurates of Lithium.—A. Duboin.—The author obtained long fine needles of $2\text{LiI} \cdot \text{HgI}_2 \cdot 6\text{H}_2\text{O}$ by leaving a saturated solution of mercuric iodide and lithium to stand during August, September, and October. He gives the full analysis of the compound, and describes the needles as flattened and striated along their length and very deliquescent. The density at 0° is 3.26. They are decomposed by water and are soluble in several alcohols, glycerin, aldehyde, acetone, and many other organic liquids. They are less soluble in nitrobenzene; insoluble in benzene and methyl iodide. *Second Iodo-mercurate*.—On cooling the above mother-liquor at 8° large crystals were found floating on the surface in the shape of small prisms. The analysis corresponded to the formula $2\text{LiI} \cdot \text{HgI}_2 \cdot 8\text{H}_2\text{O}$. The density was 2.95, and in other respects the crystals behaved as those of the preceding compound.—*Comptes Rendus*, cxli., No. 24.

A New Compound: Fluoride of Bromine, BrF_3 .—Paul Lebeau.—The author has isolated this compound (the existence of which has been recognised by M. Moissan), obtained by the action of fluorine on bromine, in a special set of apparatus consisting of glass and platinum. The composition of the compound was established by a study of its action on water and alkaline solutions. It is a colourless liquid, fuming in the air and giving off very irritating vapours. Its point of fusion lies between 4° and 5° . In the solid state it consists of a mass of beautiful colourless prisms. The chemical activity is great; a crystal of iodine thrown on to the surface of solid fluoride cooled below -10° leaves a yellow stain, then brown, and as the temperature rises the action becomes more vigorous, and the pentafluoride of iodine of M. Moissan and vapour of bromine are formed with incandescence. Sulphur in contact with the solid fluoride is inactive, but on melting it burns and gives a mixture of fluoride and bromide of sulphur. Red phosphorus, arsenic, antimony, boron, silicon react in the cold, while with carbon it has to be heated gently to start the reaction. It reacts with most of the metals and on many of their compounds. With organic compounds it behaves like fluorine. *Conclusions*.—Fluorine and bromine combine directly, giving a compound BrF_3 , a liquid which is colourless and giving a fusible solid at 4° ; it behaves chemically like fluorine.—*Comptes Rendus*, cxli., No. 24.

MEETINGS FOR THE WEEK.

TUESDAY, 23rd.—Royal Institution, 5. "Impressions of Travel in China and the Far East," by Prof. Edward H. Parker, M.A.

WEDNESDAY, 24th.—Society of Arts, 8. "The Planting of Waste Lands for Profit," by Dr. J. Nisbet.

THURSDAY, 25th.—Royal Institution, 5. "Shakespeare," by the Rev. Canon Henry Charles Beeching, M.A. & Society of Arts, 8. (Howard Lectures). "High Speed Electrical Machinery, with special reference to Steam Turbine Machines," by Prof. Silvanus Thompson, F.R.S.

FRIDAY, 26th.—Royal Institution, 9. "Walter Pater," by Arthur C. Benson, M.A.

SATURDAY, 27th.—Royal Institution, 3. "The Church in France," by J. E. C. Bodley.

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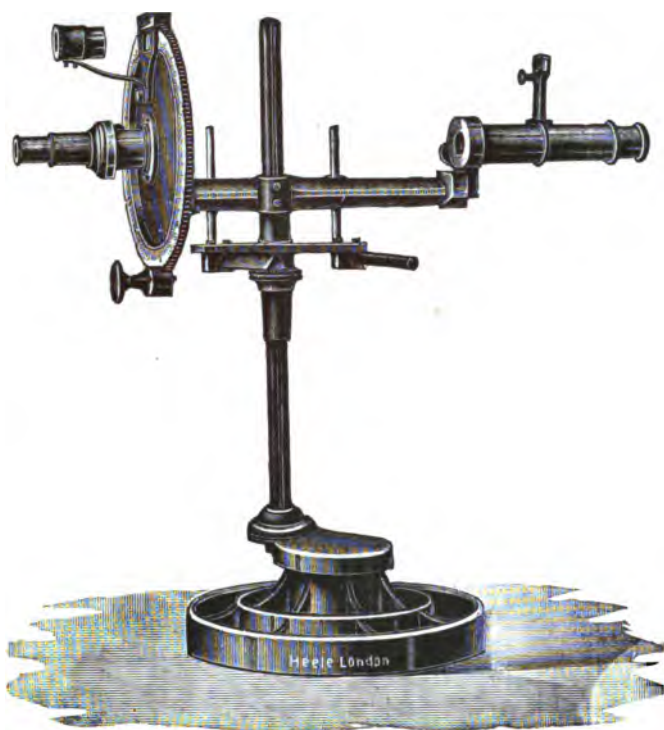
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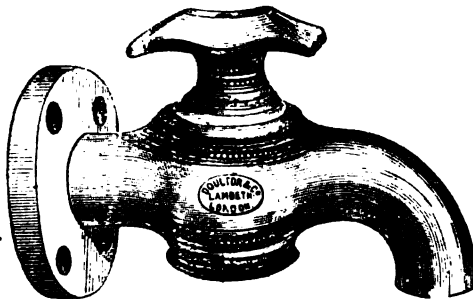
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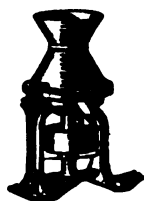
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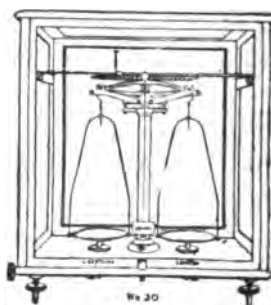
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NEW RESEARCHES ON THE ATOMIC WEIGHT OF NITROGEN.*

By PHILIPPE A. GUYE.

(Concluded from p. 24).

THE lowering of the atomic weight of silver is now made manifest by a new cycle of calculations, of which it is advisable to indicate the principle and the primary applications.

When the value of an atomic weight is to be fixed, a gravimetric ratio between the atomic weight sought and an atomic weight supposed known is made use of. For the latter, one of the atomic weights of Stas' cycle is generally taken, without any regard to the accuracy of such a weight.

The latter being based on oxygen by three atomic ratios (except silver, which is by two), it is forgotten that their accuracy cannot exceed $\pm 3/10000$ of their value ($2/10000$ for silver), and that the atomic weight sought, which results from a fourth gravimetric ratio, is only correct to $4/10000$, i.e., to $1/2500$; nevertheless, the majority of experimenters calculate, for example, atomic weights such as those of gold (206.960), mercury (200.045), platinum (194.917), &c., to three decimal places, as if correct to $\pm 1/20000$!

Thanks to physico-chemical methods, a process of calculation can to-day be evolved much more logical and more accurate, which consists in only considering as accurately known the atomic weights determined, on the one hand, by physico-chemical methods, and on the other by gravimetric ratios referred directly to oxygen.

Actually three atomic weights satisfy this double condition, those of hydrogen, carbon, and nitrogen, of which the values are—

$$C = 12.002, N = 14.009, H = 1.0076,$$

accurate to $1/10000$, approximately.

It is to be hoped that the subsidiary work will rapidly increase the number, and that soon the elements S, Cl, P will be added to this list.

However that may be, we can start from the above three elements; from them we can revise the gravimetric ratios formerly determined to refer the elements C, N, H to silver, using them to resolve the inverse problem, that is to fix the atomic weight of silver.

The numerical values of those gravimetric ratios have been carefully calculated by Clarke ("Re-calculation of the Atomic Weights, 1897, Washington"), treating the experimental data of different experimenters by the method of the least squares.

For a first application we may be content with these values.

With these data, the following ratios are obtained:—

I. Ratio Ag : AgNO₃.

According to the experiments of Penny, Marignac, Stas, and Hardin (30 determinations),—

$$100 \text{ p Ag} = 157.479 \text{ p AgNO}_3.$$

Putting NO₃ = 62.009, we have—

$$\text{Ag} = 100 \times \frac{62.009}{57.479} = 107.882.$$

II. Ratio Ag : CH₃COOAg (Silver Acetate).

According to the experiments of Liebig and Redtenbacher, Marignac, and Hardin (20 determinations),—

$$100 \text{ p CH}_3\text{CO}_2\text{Ag} = 64.636 \text{ p Ag}.$$

Putting CH₃.CO₂ = 59.027, we have—

$$\text{Ag} = 59.027 \times \frac{64.636}{35.364} = 107.886.$$

III. Ratio Ag : C₇H₅O₂Ag (Silver Benzoate).

According to Hardin's 10 determinations,—

$$100 \text{ p C}_7\text{H}_5\text{O}_2\text{Ag} = 47.125 \text{ p Ag}.$$

Putting C₇H₅O₂ = 121.052, we have—

$$\text{Ag} = 121.052 \times \frac{47.125}{52.875} = 107.888.$$

Recapitulating, we have:—

TABLE XVI.

	Atomic weight, Ag
Ratio Ag : AgNO ₃	107.882
" Ag : CH ₃ CO ₂ Ag	107.886
" Ag : C ₇ H ₅ O ₂ Ag	107.888
Mean	107.885

This result calls for certain remarks.

In the first place, it will be recognised that three independent methods to fix one atomic weight have rarely—perhaps never—given such a degree of agreement. The extreme divergence is scarcely $6/100000$, whilst between the different determinations of Stas this difference is of the order $1/5000$. Besides, it would have sufficed to take C = 12.001 instead of C = 12.002 in order to reduce the above numbers to 107.882, 107.883, 107.882.

In the second place, it is to be noticed that the first ratio is almost a direct ratio to oxygen, the group NO₃, supposed known, containing about 76 per cent of this element. Further, the extreme divergence of the different values obtained for the atomic weight of nitrogen is ± 0.005 with reference to the mean 14.009, i.e., less than $1/10000$, referring it to the weight of the group NO₃ = 62.009. From this fact the atomic weight Ag = 107.882 is correct to $\pm 1/10000$. Attributing, finally, as before, a precision of $\pm 1/10000$ to the gravimetric ratio Ag : AgNO₃, we conclude that the atomic weight Ag = 107.882 is correct to $\pm 2/10000$ in the case in which the errors add together. In other words, the atomic weight of silver cannot, in any case, exceed 107.90, nor be less than 107.86.

If, finally, one compares the mean 107.885 with the generally admitted value 107.93, the divergence is 0.055, i.e., about $1/2000$. This result is full of meaning, for a very large number of atomic weights are determined with reference to silver; if it is confirmed, the present table of atomic weights will have to be almost completely revised. I admit that it is with some hesitation that I venture to formulate this conclusion. It is so unexpected that it seems good to add some formal reservations, until a deeper investigation of the question has been accomplished.

In order to give a more complete idea of the new cycle of calculation to apply to the atomic weight of silver, it is useful to indicate, but with many more reservations, the values to which lead the incompletely controlled physico-chemical atomic weights of the elements Cl, S, P.

Their values, as calculated by various physico-chemical methods, are:—

	Cl.	S.	P.
Leduc	35.470	32.056	30.975
D. Berthelot	35.479	32.058	30.978
Guye	35.476	32.065	30.978
Mean	35.475	32.060	30.977

I would repeat that these numbers are far from affording the same scientific value as those obtained for O, H, and

* From the *Bulletin de la Société Chimique de Paris*, 1905.

N. Each of them has been merely furnished from observations relative to a single gas, the densities of the compounds SO_2 , HCl , PH_3 ; one of these densities only (SO_2) has been checked by two observers; finally, we possess for none of the elements S, P, Cl the gravimetric control of a good ratio to oxygen. Here, therefore, there are important depths to fathom by independent researches, which will be able to modify more or less the above means.

With these reservations, we now give the values of the atomic weight of silver obtained from these means, using, as before, the values of the gravimetric ratios re-calculated by Clarke.

IV. Ratio Ag : NH_4Cl .

According to the experiments of Pelouze, Marignac, and Stas (26 determinations),—

$$100 \text{ p Ag} = 49.598 \text{ p } \text{NH}_4\text{Cl}.$$

Putting $\text{NH}_4\text{Cl} = 53.514$, we have—

$$\text{Ag} = 100 \times \frac{53.514}{49.598} = 107.895.$$

V. Ratio of Chlorine to Silver Ag : Cl.

According to the latest determinations of Richards, already quoted (10 determinations),—

$$100 \text{ p Ag} = 32.867 \text{ p Cl}.$$

Putting $\text{Cl} = 37.475$, we have—

$$\text{Ag} = 100 \times \frac{37.475}{32.867} = 107.932.$$

VI. Ratio Ag_2 : Ag_2S .

According to the experiments of Dumas, Stas, and Cooke (17 determinations),—

$$100 \text{ p Ag} = 114.858 \text{ p } \text{Ag}_2\text{S}.$$

Putting $\text{S} = 32.060$, we have—

$$\text{Ag} = \frac{100}{2} \times \frac{32.060}{114.858} = 107.884.$$

VII. Ratio Ag_2 : Ag_2SO_4 .

According to the experiments of Struve and Stas (12 determinations),—

$$100 \text{ p } \text{Ag}_2\text{SO}_4 = 69.205 \text{ p Ag}.$$

Putting $\text{SO}_4 = 96.060$, we have—

$$\text{Ag} = \frac{96.060}{2} \times \frac{69.205}{30.795} = 107.928.$$

VIII. Ratio Ag_3 : PO_4Ag_3 .

According to 2 determinations of Van der Plaats, on 1 grm. of silver phosphate,—

$$100 \text{ p } \text{PO}_4\text{Ag}_3 = 77.313 \text{ p Ag}.$$

Putting $\text{PO}_4 = 94.977$, we have,—

$$\text{Ag} = \frac{94.977}{3} \times \frac{77.313}{22.687} = 107.888.$$

Recapitulating the values given by the Ratios IV. to VIII., we have:—

TABLE XVII.

Ratio	Ag		Ag.
Ag : NH_4Cl	..	..	107.895
Ag : Cl	..	..	107.932
Ag_2 : Ag_2S	..	..	107.884
Ag_2 : Ag_2SO_4	..	..	107.928
Ag_3 : PO_4Ag_3	..	..	107.888
Mean	..	..	107.905

This mean only differs from the preceding one by $\frac{2}{10000}$.

Although the values of this second series are less in agreement than those of the first, for the reasons given above, the extreme values 107.932 and 107.888 only differ among themselves by 0.044; they agree among themselves

more than the extreme values of the means calculated by Clarke, 107.907 and 108.194 (or a total of seven means, the mean of which is 107.924), thus presenting a divergence of 0.287, which is six times greater than the preceding.

Combining the two means 107.885 and 107.905, with a double weight for the first, we get to the final value—

$$\text{Ag} = 107.892,$$

or, in round numbers,—

$$\text{Ag} = 107.89 \text{ or } \text{Ag} = 107.9,$$

which represents the probable atomic weight of silver deduced from the system of atomic weights C, N, H, Cl, S, P, to which we have been led.

I shall return later to this question. The preceding exposition will suffice to make manifest the interest attaching to a revision of the atomic weights according to a new principle consisting of a combination of physico-chemical and gravimetric results.

The first application of this principle, of which I have just submitted the results, certainly shows that a new and fruitful path is opening before us. It will allow of using in a much more rational manner the valuable materials accumulated for nearly a century by all the scientists who have undertaken the determination of gravimetric atomic ratios. I have a secret hope that this work, and that of Stas in particular, may finally appear much more accurate than to-day.

P.S.—Since the assembling of this Conference certain new observations give further precision to the preceding conclusions. I will summarise them in a few words.

1. The experiments executed in my laboratory, in co-operation with Davila, confirm to a few ten-thousandths the value of the density of nitrogen dioxide found by Gray; these experiments, in process of completion, will be published shortly.

2. The critical constants of ammonia gas have been revised in my laboratory by A. Jaquerod.

Applying the method by reduction of the critical elements to the weight of a normal litre of this gas, as determined by Pintza and myself, the molecular weight of NH_3 is found to be—

$$M = 17.035,$$

whence—

$$N = 17.035 - 3.023 = 14.012,$$

a value which is within the limits previously found by physico-chemical methods.

3. Dixon and Edgar have just determined the atomic weight of chlorine by the ratio Cl : H, and have found (for $\text{H} = 1.0076$ or $\text{O} = 16$) $\text{Cl} = 35.463$, a result from which it is to be concluded that the density of the gas HCl , determined by Leduc, is probably a little high, and ought consequently to be revised.

Calculating provisionally with this value, $\text{Cl} = 35.463$, the atomic ratios iv. and v., which give the atomic weight of silver, Table XVII. ought to be revised as follows:—

Methods.	Ag.
Ratio Ag : NH_4Cl	 107.871
Ag : Cl	 107.895
Ag_2 : Ag_2S	 107.884
Ag_2 : Ag_2SO_4	 107.928
Ag_3 : Ag_3PO_4	 107.888

The mean is 107.892. If the value furnished by the ratio Ag_2 : Ag_2SO_4 be excluded, the mean is—

$$\text{Ag} = 107.886,$$

which is identical with that furnished by the more exact ratios of Table XVI., i.e.,—

$$\text{Ag} = 107.885.$$

The atomic weight of silver, obtained by starting with the elements C, H, N, Cl, S, P, from the mean of seven independent atomic ratios, is therefore contained between

107·871 and 107·895. Its exact value apparently ought not to exceed—

$$\text{Ag} = 107·89.$$

From this is evident the desirability of establishing definite and concordant values of the atomic weights of Cl, S, P by physico-chemical and gravimetric methods.

4. The lowering of the atomic weight of silver will involve the lowering of a great number of atomic weights. It would be premature to express an opinion as to the number, but it is already obvious that the result will be to increase greatly the agreement of the gravimetric ratios. I will quote as an example the ratio $\text{NaCl} : \text{NaNO}_3$. With $\text{Ag} = 107·885$, $\text{Cl} = 35·463$, $\text{N} = 14·009$, and $\text{Na} = 22·998$ (Richards' recent value $\text{Na} = 23·008$ for $\text{Ag} = 107·93$, reduced to $\text{Ag} = 107·885$), we calculate—

$$100 \text{ NaCl} = 145·408 \text{ NaNO}_3,$$

whilst the mean deduced by Clarke from 23 determinations by Penny, Stas, and Hibbs give—

$$100 \text{ NaCl} = 145·418 \text{ NaNO}_3.$$

At 1/15000 about, the limit of accuracy of the experiments, the two values agree.

This is a confirmation of the new value of the atomic weight of silver, which is the subject of the preceding considerations.

THE THERMO-CHEMICAL RELATIONS OF CARBON, HYDROGEN, AND OXYGEN.

By JOHN CLARK THOMLINSON, B.Sc. (Dunelm).

A CONSIDERATION of the thermo-chemical data available to the physical-chemist has lead to the following results in an attempt to determine the heats of combination of the elements carbon, hydrogen, and oxygen.

The formation of methane, CH_4 , and ethane, C_2H_6 , takes place with the evolution of 21·750 calories and 28·560 calories respectively, and assuming that the heat of combination of carbon and hydrogen is the same in both reactions we obtain as a result $\text{C} = -8·130$ calories, $\text{H} = 7·470$ calories, carbon having a negative value corresponding to an actual absorption of heat during reaction.

To confirm the assumption we have just made, we find on applying these values to the higher hydrocarbon propane, C_3H_8 , of this series that the calculated heat of formation is 3·537 calories, closely agreeing with the number 3·511 calories obtained by experiment.

Hence we may say in the compounds under consideration that the mode of union of the carbon atoms in ethane and propane in no way affects the thermal value of that element.

Methane, ethane, and propane are, however, all saturated compounds; when we take into consideration the heats of formation of the corresponding unsaturated hydrocarbons, we find we have to assign a different value to the heat of combination of carbon in such groups as $\text{C}=\text{C}$, $\text{C}\equiv\text{C}$.

Ethylene, C_2H_4 , is formed with the absorption of 2·710 calories; acetylene, C_2H_2 , has a heat of formation = -48·170 calories. If hydrogen still retains its value 7·470 calories, then $\text{C}=\text{C} = -16·300$ calories, the symbol $\text{C}=\text{C}$ indicating a carbon atom united by two bonds of union to another carbon atom, and similarly in acetylene $\text{C}\equiv\text{C} = -31·560$ calories.

We have assumed here that hydrogen has the same heat of combination in unsaturated as in saturated hydrocarbons; the following thermo-chemical relations will support these conclusions:—

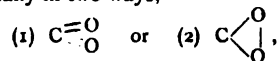
When carbon unites with oxygen to form carbonic oxide and carbon dioxide, a much greater evolution of heat takes place in the latter reaction, as is evident from the following equations:—

$$\text{I. } 2\text{C} + 2\text{O} = 2\text{CO} + 58·000 \text{ calories}$$

$$\text{II. } \text{C} + 2\text{O} = \text{CO}_2 + 96·960 \quad ,,$$

the heat of formation of one molecule of carbon dioxide being nearly double that of two molecules of carbonic oxide.

Further, the formula of carbon dioxide can be represented graphically in two ways,—



in which carbon is respectively quadri- and di-valent.

We should expect, as will be shown later is the case, that oxygen combining with both its bonds of union to another atom would cause a greater evolution of heat than the union of the group —O—O— , and are hence justified in regarding Formula 1, in which carbon is quadrivalent, as the true structural formula for carbon dioxide.

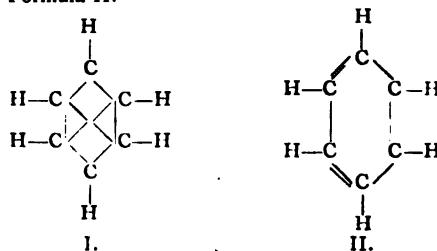
To return now to the formation of dioxide and monoxide, we find carbon quadrivalent in Equation II., whereas in Equation I. it is evidently divalent, and as the nature of the union of the oxygen in both compounds is the same, one quadrivalent carbon atom in uniting with two oxygen atoms gives rise to an evolution of 38·960 calories in excess of that due to two carbon atoms uniting with two atoms of oxygen to form two molecules of carbon monoxide, and since we have assigned to quadrivalent carbon a heat of combination -8·130 calories, that of di-valent carbon is -8·130 - 19·480 calories, i.e., is equal to -27·610 calories.

A comparison of these values for the heat of combination of carbon with those obtained for it in the case of ethylene and acetylene will show us that in these compounds carbon may be regarded as respectively tri- and di-valent.

For quadrivalent carbon $\text{C}\equiv\text{C} = -8·130$ calories, and when di-valent carbon = -27·610 calories, the mean of these numbers representing tri-valent carbon if such exists as having a heat of combination = -17·870 calories, and the value obtained for the carbon atom in ethylene is $\text{C}=\text{C} = -16·300$ calories.

Similarly, $\text{C}\equiv\text{C} = -31·560$ calories in acetylene and the value for divalent carbon as in carbonic oxide has been found to be -27·610 calories, giving as the experimental and calculated values (calculated from the latter value for divalent carbon), -48·170 calories and -40·280 calories respectively, which numbers although showing a difference of about 20 per cent is an approximation in favour of viewing carbon as having a di-valent value in acetylene.

In this connection it is interesting to note that in benzene, C_6H_6 , the heat of formation, taking the ordinary number for the heat of combination of hydrogen $\text{H} = 7·470$ calories, gives a value for carbon $\text{C} = -7·655$ calories, so that this compound may be looked upon as being essentially a saturated hydrocarbon, in so far as its thermal value is concerned, corresponding to Formula I., which we should regard as more nearly indicating a condition of saturation than Formula II.



We see, then, that carbon must be looked upon as having different thermal values according to the manner in which it is combined; let us now return to Equation I.,—

$$\text{C} + \text{O}_2 = \text{CO}_2 + 96·960 \text{ calories (I.)},$$

which gives a value for the heat of combination of oxygen $\text{O} = 52·545$ calories, with the ordinary value for quadrivalent carbon, which, with the numbers we have obtained for hydrogen, gives as the calculated heat of formation of water 67·485 calories.

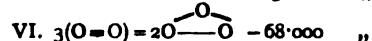
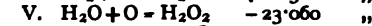
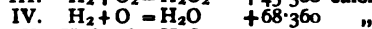
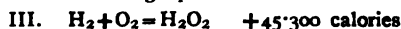
Now, water has been found experimentally to have a heat of formation 68·360 calories for the liquid and 59·133 calories for the gaseous product at 18—20° C.

We have hitherto been dealing with the latter conditions, and here the calculated and experimental values again show approximation, though truly not very closely, the difference being about 15 per cent.

We have, however, shown that we can obtain approximate values for the heats of combination of carbon, hydrogen, and oxygen, giving results for the heats of formation of compounds which show an agreement with those obtained by experiment that would lead to our assuming that such are approximately the conditions actually occurring during the formation of the compounds of these elements.

Further examples supporting those already given are those of hydrogen peroxide and ozone, which are formed from water and oxygen respectively with absorption of heat.

Compare the following equations :—



If $\text{H}-\text{O}-\text{O}-\text{H}$ be taken as graphic representation of the formula for hydrogen peroxide, we find the heat of combination of the group $-\text{O}-\text{O}-$ is equal to 30·360 calories, so that the entrance of a second atom of oxygen into the molecule of water would, according to the value we have already obtained for the heat of combination of an oxygen atom, $\text{O} = 52\cdot545$ calories, be accompanied by an absorption $52\cdot545 - 30\cdot360$ calories of 22·185 calories; that actually taking place as found by experiment is 23·060 calories.

Here we have the double bond of an oxygen atom in union with atoms of other elements broken up in such a way that one of the bonds becomes united with that O_1 another atom of oxygen, this action having a thermal value = -23·060 calories.

In the reaction represented by Equation VI. three molecules of oxygen are converted into two molecules of ozone, 68·000 calories being absorbed, and here the same action as in the case of hydrogen peroxide takes place, the splitting up of the double oxygen bond by union with a further atom of oxygen taking place in a threefold sense, and calculating from the value found in the case of hydrogen peroxide, we should expect a heat absorption equal to 69·180 calories; that actually taking place is 68·000 calories.

The heat of formation of amylene, C_6H_{10} , is 19·000 calories where the product is liquid; that obtained by calculation—and here, as in the following examples, the calculated values refer to gaseous products—is 17·710 calories.

Further examples are given in the following table :—

Compound.	Heat of formation determined experimentally.		Heat of formation by calculation.
		Calories.	Calories.
Acetic acid, $\text{C}_2\text{H}_4\text{O}_2$..	..	120°340	118°430
Propionic acid, $\text{C}_3\text{H}_6\text{O}_2$..	..	109°400	125°520
Oxalic acid, $\text{C}_2\text{H}_2\text{O}_4$..	..	202°540	208°860
Cane-sugar, $\text{C}_{12}\text{H}_{22}\text{O}_{11}$..	..	601°500	644°775

In all these cases the numbers are greater when calculated than are found by experiment, in spite of the remarks made in the case of amylene, but considering what we have already found to be the nature of the union of carbon, hydrogen, and oxygen, we may find an explanation of this in the constitution of the compounds themselves.

Other examples might be quoted, but such thermo-chemical relations as we have hitherto studied may suffice to confirm us in considering certain values as at any rate approximately representing the heats of combination of the elements carbon, hydrogen, and oxygen.

This paper has been written with a view to bringing forward the thermo-chemical relations which have led to a

belief in such heats of combinations, and in such a branch of the science, where so many attendant phenomena, physical and otherwise, may affect the data we have to work with, what is only approximately true may be of value in elucidating the problems of thermo-chemistry.

THE CHEMICAL SEPARATION OF THE RADIO-ACTIVE TYPES OF MATTER IN THORIUM COMPOUNDS.*

By HERMAN SCHLUNDT and RICHARD B. MOORE.

(Concluded from p. 28).

The Theoretical Curves.

THE foregoing explanation for the initial peculiarities of the curves of recovery and decay finds further confirmation in the theoretical curves which are obtained on the basis of the suppositions made. The method of treatment here employed follows, in the main, the exposition given by Rutherford ("Radio-activity," 1904, pp. 268—272, 297—300) and by Stark ("Jahrbuch der Radioaktivität," 1904, i., 1). According to the disintegration theory, a constant proportion of the atoms of a radio-active element is continuously undergoing change, and the ionisation produced may be used for ascertaining the fraction of the atoms which are changing per second. This constant rate, called the *radio-active constant*, is the fundamental physical characteristic of the different radio-active types of matter. It has a different value for each type of active matter, but is a definite constant for each particular type. The radio-active constant for ThB is 0·0125, when the time is expressed in minutes, which indicates that ThB is continuously undergoing change at the rate of 1·25 per cent per minute. The ionisation produced by a particular type of radio-active matter depending, as it does, upon the number of atoms present and their rate of change, is directly proportional to the product of the rate and the number of atoms present. This law of radio-active change is the same as the law followed by a mono-molecular chemical reaction, the inversion of cane-sugar for example, and both laws are illustrations of the more general law of nature which Lord Kelvin has happily termed the "Compound Interest Law," which states that the amount of change in a given time is directly proportional to the quantity present in the system (Mellor, "Higher Mathematics for Students of Chemistry and Physics," p. 40).

The rate of change of a radio-active element is expressed by the equation—

$$\frac{dn}{dt} = -rn;$$

where n denotes the number of atoms present at the time t , and r the radio-active constant, the minus sign indicating that the change causes a decrease in the quantity of the active element. By integration of the above equation,—

$$n = n_0 e^{-rt};$$

where n_0 denotes the number of atoms considered at the outset, when $t=0$. The activity, I , of the sample at the time t , is given by—

$$I = rn = rn_0 e^{-rt},$$

where c is the proportionality constant. The values of the theoretical curve of decay of ThX (Plate I.), were calculated from the above equation. The radio-active constant of ThX has the value 0·0072 when t is expressed in hours (Rutherford, "Radio-activity," p. 298).

When the radio-active substance produces another, which is also radio-active, then the activity at any time is proportional to the sum of the atoms of each which are undergoing change. The curve showing the progress toward equilibrium will then frequently show irregularities.

* From the *Journal of Physical Chemistry*, ix., No. 8, p. 62a.

The residue obtained in the fumaric acid separation contains ThX and ThA just after separation, but ThA at once produces ThB, which changes rapidly into a final inactive product. The successive changes occurring simultaneously in the residue are:—

ThX $\rightarrow$ Emanation $\rightarrow$ ThA $\rightarrow$ ThB $\rightarrow$ Final Product.

The activity of the solid residue itself is due to ThX and ThB. In the consideration which follows, the second product, the emanation, is not introduced, since its rate of transformation is very rapid in comparison with the other products.

If n , n_1 , n_2 denote the number of atoms of ThX, ThA, and ThB present at the time t after separation, and r , r_1 , and r_2 the corresponding radio-active constants, then the activity I is expressed by

$$I = rn + k r_2 n_2 \dots \dots \dots (1),$$

where k denotes the ratio of the ionisation of ThB to ThX. The radio-active constants have the values, $r = 0.0072$; $r_1 = 0.063$; $r_2 = 0.75$, when t is expressed in hours. The value of n is obtained from the formula—

$$n = n_0 e^{-rt}.$$

The value of n_2 depends upon the value of n_1 , and is determined by the following considerations: Assuming that ThX and ThA are in equilibrium immediately after the separation, then the quantities of each present are inversely proportional to their radio-active constants; that is,—

$$n : n_1 = r_1 : r.$$

Introducing the values of r and r_1 we have—

$$n_1 = \frac{n}{8.75} = \frac{n_0}{8.75} e^{-rt}.$$

Now, the number of atoms of ThB continuously undergoing change is $r_2 n_2$, and the number produced from ThA at the same time is $r_1 n_1$. Hence the rate of change in ThB,—

$$\frac{dn_2}{dt} = -r_2 n_2 + r_1 n_1,$$

whence

$$\frac{dn_2}{dt} + r_2 n_2 = \frac{r_1 n_0}{8.75} e^{-rt}.$$

Integration of this equation gives—

$$n_2 = \frac{r_1 n_0}{8.75 (r_2 - r_1)} (e^{-rt} - e^{-r_2 t}).$$

The theoretical values in the following table were calculated from Formula 1. The value of $k = 0.5$ used, was obtained from the ratio of the ordinates of the activities of ThB and ThX at the end of the second day. The experimental values are given in the second and fourth columns of the table, whilst the first column gives the time which had elapsed since the separation. (See also Curves B and C, Plate I.).

Time.	Fumaric acid.	Theoretical value.	Pyridine.
0	100	100	100
20 minutes	—	111	—
30 "	116	115	108
60 "	129	127	113
2 hours	147	137	127
4 "	155	144	141
5 "	158	145	142
6 "	158	144	142
8 "	154	142	140
16 "	—	134	—
24 "	128	127	122
2 days	106	107	103
4 "	74	75	73
8 "	37	38	36
12 "	18	19	17
16 "	—	9.5	—
20 "	5.6	4.7	4.5

The times at which the theoretical and experimental values attain a maximum are practically the same, thus confirming the assumptions made. The variations of the experimental from the theoretical values is greater at the outset than the experimental error, but these differences may be accounted for by assuming that the ionisation ratio of the two radio-active types of matter varies, gradually assuming a smaller value, which is quite probable in view of the fact that the matter causing imparted activity is formed by the emanation, and the emanating power varies considerably with the nature and physical condition of the substance. Moreover, the presence of the emanation introduces experimental difficulties.

Considering now the ammonia separation, it was shown by Rutherford and Soddy that the filtrate residue simply contains ThX immediately after the separation, but ThX at once produces the inactive ThA, which, in turn, produces ThB, the last active product. This case differs from the one just considered, inasmuch as no ThA is present at the outset. Following the same notation as in the previous case, it is seen that n , the number of ThX atoms present at the time t , is obtained from $n = n_0 e^{-rt}$.

The rate of change in ThA is expressed by—

$$\frac{dn}{dt} = -r_1 n + rn,$$

which, upon integration, gives—

$$n_1 = \frac{r n_0}{r_1 - r} (e^{-rt} - e^{-r_1 t}).$$

The rate of change in ThB is expressed by—

$$\frac{dn_2}{dt} = -r_2 n_2 + r_1 n_1.$$

This equation, after substituting in it the value of n_1 when integrated, gives—

$$n_2 = \frac{r r_1 n_0}{r_1 - r} \left(\frac{e^{-rt}}{r_2 - r} - \frac{e^{-r_1 t}}{r_2 - r_1} \right) + \frac{r_1 r n_0}{(r_2 - r)(r_2 - r_1)} e^{-rt}.$$

The activity at the time t is given by—

$$I = rn + K r_2 n_2,$$

where K again represents the ratio of ionisation due to ThB.

The theoretical values in the table below were calculated from the last equation. In the calculation of the values of n_2 , the last term in the equation becomes negligible after the fourth hour. The value of $K = 0.44$, here employed, is the one given by Rutherford ("Radio-activity," p. 298).

Time.	Theoretical values.	Experimental values.
0	100	100
1 hour	100+	101
5 hours	105	106
10 "	111	110
16 "	116	117
20 "	115	117
24 "	114	113
50 "	103	100
3 days	89	88
4 "	75	73
8 "	37.7	38
12 "	18.8	19
20 "	4.7	5

Here again the maxima in the theoretical and experimental values practically coincide, and the theoretical and experimental values show a close agreement throughout.

Removal of ThX by means of other Reagents.

Various qualitative tests were made with other reagents which precipitate thorium quantitatively from aqueous solutions of its salts. In many cases the residue obtained by evaporating the filtrate and igniting it was highly radio-active, showing that a radio-active constituent had been removed. The following additional reagents remove some

form of radio-active matter from thorium compounds: aniline, benzoic acid, meta-nitrobenzoic acid, tannic acid, potassium xanthogenate, hydrogen peroxide, piperidine, phenylhydrazine. In fact, the major part of the reagents tried, which precipitate thorium completely, yielded radio-active residues. The precipitates were also radio-active, thus showing that a non-separable portion of the activity is either due to thorium itself or to a new type of matter closely resembling thorium in its behaviour towards reagents. The distribution of the radio-active components between the precipitate and residue, when the above reagents are used, is being studied, and in this connection the separations are being conducted with a sample of thorium nitrate which has been specially purified.

Although no very accurate tests on the emanating power of the different thorium precipitates were carried out, still the values observed show that the different compounds vary considerably in this respect. Rutherford and Soddy first directed attention to this point (*Journ. Chem. Soc.*, 1902, lxxxi., 851). They found that when thorium was precipitated apparently under the same conditions the precipitate often showed marked difference in emanating power. The thorium oxide obtained by us by means of pyridine generally possessed less emanating power than the oxide obtained by means of ammonia. The thorium fumarate precipitate possessed a high emanating power. Although it varied considerably it was usually more than twice that of the ammonia precipitate. Hence thorium fumarate exceeds in emanating power any of the other thorium compounds thus far examined.

Summary of Results.

1. The separation of the radio-active types of matter in thorium compounds by chemical methods was studied from a qualitative standpoint, and a detailed quantitative study was carried out with two reagents—pyridine and fumaric acid.
 2. The removal of ThX from thorium compounds by means of ammonia, discovered by Rutherford and Soddy, was confirmed. Our experiments constitute an extension of the method of separation employed by these investigators.
 3. Pyridine and fumaric acid remove from thorium nitrate solution ThX and the inactive product, ThA, of the matter which constitutes the imparted activity of thorium. The precipitate contains the non-separable activity and the radio-active component of the imparted activity, ThB, which decays to half value in fifty-five minutes.
 4. It was shown both by experimental and theoretical considerations that the presence of ThA in the filtrate residue, in the separation by means of fumaric acid, and its presence in the precipitate when ammonia is employed, accounts for the initial differences observed in the curves of decay and recovery of the separated products.
 5. ThA was isolated from the other radio-active products by chemical methods. Pure ThB was isolated by electrolysis.
 6. Inactive thorium was not obtained in the course of the experiments.
 7. One precipitation of an aqueous solution of thorium nitrate with fumaric acid yields a thorium precipitate whose activity at the end of five hours is less than one-fourth the final activity of the product.
 8. The emanating power of thorium fumarate was found to be approximately double that of the thorium oxide obtained by precipitation with ammonia, while the emanating power of the oxide obtained in the pyridine precipitation fell below that obtained by the use of ammonia.
- Further experiments on the separation of the radio-active products in thorium compounds by chemical methods are in progress.

Correction.—In the Obituary Notice in our last issue (p. 28), the date of the decease of the late Dr. Sprengel was incorrectly given as Friday, January 12th, instead of Sunday, January 14th.

MECHANICAL ANALYSIS OF SOILS.

A SUGGESTION FOR A LONG TUBE SEDIMENTATION PROCESS.

By J. ALAN MURRAY.

THE importance of finding some quick and handy method of "sorting out the particles of soil into groups each of which contains material lying between specified limits of size" is generally recognised. The method of Hall and Luxmore revised by Hendrick described in a recent paper, whatever its merits in other respects, cannot be described as either quick or handy. To effect a complete separation of the finer from the coarser particles by mixing the soil with water and decanting off the portion which remains in suspension after standing some time, requires repeated washing of the sediments. This occupies a long time, and involves the accumulation of much water which must be subsequently evaporated.

The repeated washing of the sediments is of course indispensable, because when the particles of soil are suspended in water and allowed to settle, some of the smaller particles near the bottom are deposited sooner than some of the larger ones which have a greater distance to travel. The depth of the column of water employed is purely arbitrary, but no variation in the depth of the column will obviate this difficulty; in fact, since the particles are more or less uniformly distributed through it, the longer the column the more incomplete will be the separation.

But if a fairly long column of water be taken—say, about a metre—and all the particles start from the top at the same time, then the largest will reach the bottom first and the others after longer intervals according to size.

To make a separation in this way it is necessary to devise some means by which a quantity of liquid containing the fine soil in suspension can be introduced at the top of such a long column of water, and by which the fractions may be removed as they collect at the bottom. In order to admit of the removal of the fractions the tube must be open at the bottom and consequently must be closed at the top. All attempts to introduce the liquid containing the soil in suspension by means of side tubes, stopcocks, &c., failed owing to the considerable downward pressure of the column of water. It is, however, quite simple to introduce the liquid at the bottom of the long tube, and then invert it. This comes to the same thing, and it was in this way that the more successful experiments were conducted.

The apparatus employed consisted of a pear-shaped flask (*i.e.*, one having a very sloping shoulder) of about 200 c.c. capacity; from this a portion of the neck was cut off, leaving a piece (about an inch and a-half) just long enough to insert through an indiarubber cork, by means of which it can be attached to a long piece of glass tubing. The tube actually used was one which happened to be in the laboratory, and which proved on measurement to be 147 c.m. long by 2.3 c.m. internal diameter. A wide shallow glass basin about 20 c.m. diameter and a few small porcelain evaporating dishes about 7.5 c.m. diameter are also necessary. It was found, however, that notwithstanding the shape of the flask there was some tendency for the finer particles to lodge on the shoulder, and it was later replaced by an Erlenmeyer's flask of which the neck, after removal of the flange, was exactly the same diameter as the tube. This was attached by means of a piece of indiarubber tubing fitted outside of the tube and of the neck of the flask.

The experiment was carried out as follows:—Five grms. of the air-dried fine soil (*i.e.*, that which had passed through 100 mesh sieve), thoroughly disintegrated in dilute ammonia solution, was put into the flask, which was then vigorously shaken, filled nearly full of water, and attached to the tube. Water was then poured gently down the side of the tube (so as not to disturb the sediment in the flask) till it also was full, when the open end was closed with a cork, and the whole inverted over the large basin of water.

EXAMPLE I.—Showing Results obtained in Three Trials with a Stiff Clay Soil, Untreated with Acid, after Rough Preliminary Separation into Coarse and Fine Material.

Fraction.	Time. Minutes.	Average diameter of particles. M.m.	Grms.			Per cent.		
			I.	II.	III.	I.	II.	III.
1.	5	0'13	0'420	0'535	0'374	8'40	10'70	7'48
2.	15	0'06	0'783	0'838	0'660	15'66	16'76	13'20
3.	40	0'03	0'923	0'980	0'952	18'46	19'60	19'04
4.	60	0'02	0'708	0'572	0'759	14'16	11'54	15'81
5.	180	0'013	0'590	0'542	0'569	11'80	10'84	11'38
6.	480	0'006	0'590	0'431	0'634	11'80	8'62	12'68
7. Residue		0'002	0'920	0'920	0'880	18'40	18'40	17'60
Moisture			0'205	0'205	0'205	4'10	4'10	4'10
			5'139	5'003	5'033	102'78	100'50	100'66

EXAMPLE II.—Showing Results obtained in Duplicate Trials with Rothamsted Soil (Plot 1 C, Barnfield, 1903), after Treatment with Acid, as recommended by Hall. No Preliminary Separation.

Fraction.	Time. Minutes.	Rate of falling. C.m. per Sec.	Average diameter of particles. M.m.	Grms.		Per cent.	
				I.	II.	I.	II.
1.	2'6	= 1 " 1	0'16	0'223	0'229	4'46	4'58
2.	13	= 1 " 5	0'05	1'545	1'523	30'90	30'46
3.	26	= 1 " 10	0'04	0'955	1'043	19'10	20'86
4.	52	= 1 " 20	0'025	0'707	0'644	14'14	12'88
5.	130	= 1 " 100	0'020	0'427	0'442	8'54	8'84
6.	260	= 1 " 400	0'010	0'203	0'114	4'06	2'28
7. Residue			0'004	0'531	0'662	10'62	13'24
Loss on solution				0'352	0'352	7'04	7'04
Moisture				0'150	0'150	3'00	3'00
				5'093	5'159	101'86	103'18

The cork closing the end was immediately withdrawn, and one of the small porcelain evaporating dishes, previously dried and weighed, placed beneath the open end of the tube.

The sediment began to descend whenever the tube was inverted, and the first particles were deposited in the dish in about fifteen seconds. After a short interval this dish, containing the first portion, was removed and replaced by another, and this again by a third, and so on. These dishes containing the several fractions of the sediment thus removed from the large basin were of course full of water, of which, however, a considerable part was separated by simple decantation, after standing for a few minutes, and the remainder driven off on the water-bath. The dried residue was then weighed, and examined under the microscope for determination of the size of the particles.

The small dishes (in which the fractions of sediment are collected) being entirely submerged in the water in the large basin, great care must be exercised in changing them, for, if the water be agitated, the fine sediment, which is easily disturbed, at once diffuses through the water, and is lost to the fraction to which it properly belongs. It was found, however, that with a little practice this operation could be easily and quickly performed without appreciable loss.

It was noticed in the course of the experiments that some of the particles falling through the column of water are caught and carried up again by ascending currents, of which the cause has not been ascertained or any means of preventing them. That they are due to some extent to the motion of the falling solid particles is probable, but it seems unlikely that this is the sole cause. Careful observation of individual particles showed that under favourable circumstances they never rose more than about $2\frac{1}{2}$ inches before they again took a downward course. Possibly a series of short downward pointing inner tubes or even ribs on the long tube would reduce the currents to a minimum.

Examination of the fractions under the microscope showed that while each consisted of particles of fairly uniform size, still some of the finest particles could be discerned among the coarsest. The finer particles may have

simply adhered to the larger ones, or they may have been drawn downwards in the wake (as sailors say) of the latter. Probably both causes combined to bring about the result. At all events the fact suggested the expediency of making a preliminary rough separation of the coarser and finer material, and this further modification was accordingly introduced in the later experiments as follows:—The fine soil after being disintegrated in weak ammonia solution was allowed to stand for fifteen minutes, and the water containing the finer material in suspension then poured off. The coarser material remaining in the beaker was then introduced into the flask, and the process carried out as previously described. Three fractions were collected. The first five minutes after the first particle had reached the bottom, the second fifteen minutes (*i.e.*, ten minutes after the first dish had been removed), and the third forty minutes from the commencement (*i.e.*, twenty minutes after the second dish had been removed). Almost the whole of the remainder of the coarser material was deposited within an hour; this, however, was not treated as a fourth fraction, but as fine material which had escaped through preliminary separation, and was accordingly returned to the beaker containing the finer material with which it was thoroughly mixed, and the whole introduced into the flask, and allowed to fall through the long column of water—the same water which had served for the separation of the coarser material being used over again so that there should be no loss of material which remained suspended in it. Three fractions were collected in this case also after one, three, and seven hours respectively, making six fractions in all. The liquid remaining in the flask and tube (containing what had not settled out in seven hours), amounting to a little over a litre, was measured, an aliquot part taken and evaporated to dryness, and a seventh and last fraction so obtained.

The value of the preliminary separation became at once apparent on examination of the fractions of the sediment, which were found to be much more homogeneous than before, though naturally a number of particles of exceptional size, above and below the mean, were always present in each. The mean or average size of the particles was,

however, readily recognisable—the same result being given repeatedly by independent observers. In fact, the mean size of the particles was so much more easily determined than the extreme limits that it has been found convenient to express the results in that form.

Should the process prove generally acceptable its utility would be greatly enhanced if a common basis for expression of results were agreed upon; that of a simple statement of percentages falling at a mean rate of so many centimetres per second has much to recommend it.

In the earlier experiments the sample of soil used was a stiff clay—it has actually been used for making bricks—which it was thought would present all difficulties likely to be met with. As it contained but little lime or organic matter, and the object at that stage was merely to test the method of separating the particles of different sizes, it was thought better to omit preliminary treatment with acid. The degree of accuracy with which the process can be carried out may be judged by comparison of the results obtained after experience had been acquired. Some of these results are given in the table, both in percentages and in actual weights of the fractions. More recently the process has been tried with a sample of Rothamsted soil (Barnfield plot, 1 C, first 9 inches, taken in 1903) after preliminary treatment with acid as recommended by Hall. The results are given in the form suggested above, *i.e.*, percentages of particles falling at a given rate per second.

This soil being so much lighter in character it was found unnecessary to make a preliminary separation into coarse and fine material.

It is difficult to compare the results with those obtained by Hall with the same soil, but it is evident that they differ considerably in regard to the proportions of the finest and coarsest material, and to a less degree in regard to the proportions of the intermediate groups. The correspondence between the duplicates is, however, fairly satisfactory.

THE TEMPERATURE OF SOLUTIONS HEATED BY OPEN STEAM.*

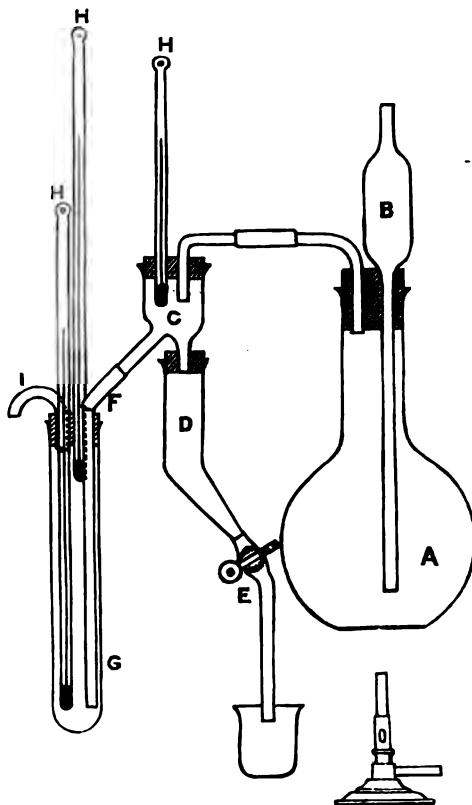
By THOS. STEEL, F.C.S., F.L.S.

THE fact that it is possible to heat solutions by means of open steam to temperatures higher than that of the steam itself does not appear to be so well known as its interest and importance warrant. I have not seen any direct reference to this point in works on physical subjects, and, indeed, am only aware of two references thereto in literature. The first allusion with which I am acquainted is in "Report of the British Association," 1869, *Trans.*, lxxv., and 1870, *Trans.*, lxiv., where Peter Spence records his accidental discovery of the phenomenon, and gives results of a series of laboratory experiments on the determination of the boiling-point of saturated solutions of various salts by blowing in steam at 100° C. Spence gives no explanation of the cause of the phenomenon, but contents himself with recording the results of his experiments. The other reference to the matter occurs in a recent publication, *Zeit. Ver. Deuts. Zucker-Ind.*, 1904, 1159, in a paper by H. Claassen on the "Boiling-point of Sugar Solutions." (*See Journ. Soc. Chem. Ind.*, 1904, 1105 and 1228). In this paper Claassen points the fact out clearly, and gives a brief theoretical explanation.

It is my purpose in the present paper to give a description of an apparatus which I have used, with details of some illustrative experiments, and to explain the theoretical considerations governing the phenomenon under discussion.

In the accompanying illustration of the apparatus used, A is a 32 oz. flask fitted with a 100 c.c. pipette; B is a safety tube; c is a tube for removing water from the steam

and allowing the temperature of the latter to be observed; D is a tube in which condensed water may gather; E is a rubber joint fitted with a spring clip, connecting to a tube leading to a small beaker placed beneath; F is a rubber joint connecting the tube which leads the steam into the large test-tube G; H, H, H, are thermometers; and I is a tube for carrying off surplus steam. When the apparatus is in use c is wrapped round with a piece of cotton-wool, while the tube G is inserted into a cylinder packed with the same insulating material. The water in the flask A is boiled, the clip at E being left open until the apparatus is warmed up and steam issues freely. As water accumulates in the tube D during the course of the experiments, it may be discharged by opening the clip. On placing a solution in the tube G and allowing the steam to pass through it, the temperature will rise steadily until a point is reached



at which it will remain for a time, and then gradually fall. If the steam be allowed to escape at any moment by opening the clip at E, and the tube G be removed with its fittings in place, and heated to boiling over a lamp, the temperature reached will, as a rule, be very little higher than that which was attained by means of the steam. In many cases, by having a quantity of the powdered salt present so as to keep the solution in a saturated state, the boiling-point of a saturated solution, or a temperature very near that point, may be readily reached and maintained. Under the conditions of my experiments the steam passing through the tube c has a temperature of 99° 0', which rises to 99° 2' or 99° 3' when subjected to the hydrostatic pressure in the tube G. The steam escaping from G has a temperature of 99° 0'. Some scraps of platinum foil or bits of porous clay pipe are in all cases placed in the solutions for the purpose of promoting steady boiling.

In the following table the temperatures attained by some non-saturated solutions, when heated by steam under the

* From the *Journal of the Society of Chemical Industry*, xxiv., No. 11.

above conditions, are stated, as also the temperatures at which the same solutions boil when heated over a lamp:—

Non-saturated Solutions.

	Boiling temperatures.	
	With steam.	Over lamp.
Disodium phosphate	103°0	103°5
Sodium carbonate	104°2	104°5
Potassium nitrate	105°5	106°5
Ammonium chloride	111°5	112°5
Sodium acetate	112°5	114°0
Sodium nitrate	117°5	118°5
Potassium acetate	147°0	152°0
Calcium chloride	149°2	150°0
Ammonium nitrate	155°0	156°0
Ammonium nitrate (stronger) ..	163°0	164°0

It will be noticed that in all cases the temperature reached by heating over the flame is somewhat higher than that attained by means of steam. The temperatures attained by the various solutions depend, of course, on the concentrations. With a strong solution of ammonium nitrate steam at atmospheric pressure is competent to raise the temperature to 163°. In carrying out this experiment steam was passed direct into moist ammonium nitrate, and the temperature mentioned was quickly reached in spite of the fact that there is a considerable cooling effect when this salt is dissolved.

Saturated solutions, as I have before mentioned, may be tested in the same way by having a quantity of the powdered salt present in the hot solution. In the next table are given the temperatures attained by the use of steam with saturated solutions of a number of salts, and, for comparison, the boiling-point of the same solutions as obtained by heating over the lamp and also as published in "Watts' Dictionary of Chemistry" and elsewhere.

Saturated Solutions.

	Boiling temperatures.		
	With steam.	Over lamp.	Boiling-points (a).
Potassium chlorate ..	103°3	103°5	104°2
Zinc sulphate ..	103°5	103°8	104°4
Sodium carbonate ..	105°2	105°5	104°6
Potassium chloride ..	108°0	108°2	108°3
Sodium chloride ..	108°0	108°2	108°4
Potassium nitrate ..	114°0	115°0	115°9
Ammonium chloride ..	114°0	115°0	114°2

(a) The boiling-point of a saturated solution of zinc sulphate is by Griffiths, and is given in "Corney's Dictionary of Chemical Solubilities," p. 459; that of potassium chloride is the figure found by Legrand and quoted in Clarke's "Rules, Tables, and Data," p. 369; all the others are given on the authority of Legrand in "Watts' Dictionary of Chemistry," vol. iii., p. 89.

As is the case with non-saturated solutions the temperatures reached by the use of steam are somewhat lower than those obtained by boiling over the lamp, while the latter figures are sometimes higher and sometimes lower than the published boiling-points for saturated solutions of the salts experimented with. In this connection it should be explained that the salts used by me were the ordinary commercial ones sold by reputable dealers for laboratory use.

When dealing with very soluble substances such as calcium chloride, the solubility of the salt prevents saturation being reached. When steam is blown into granular calcium chloride, a solution is formed having a temperature of 149°2, while by heating over the flame this solution boils at 150°. If a saturated solution of the same salt is prepared by stirring powdered calcium chloride into the above solution kept boiling over a flame, the pasty mass formed boils at 175°. If now the tube containing the hot saturated solution be quickly placed in the lagged holder, and

steam passed in, the solution does not remain at the same temperature, but steadily cools. If the steam be now turned off, the contents of the tube allowed to cool somewhat, and steam again turned on, the temperature rises to a few degrees below the point at which it stood in the first instance. With ammonium chloride, treated in the same manner, the steam merely passes through and does not form a solution, consequently the contents of the tube do not rise above the temperature of the steam itself. On adding a little water, a saturated solution is formed which becomes heated to a temperature of 114°0, or only 0°2 less than the boiling-point for a saturated solution of this salt given in "Watts' Dictionary," while on heating over the lamp it boils at 115°0, which is 0°8 higher. A saturated solution of zinc chloride is stated in "Watts' Dictionary" to boil at 172°. With the apparatus described it is possible, by starting with the powdered salt into which steam is passed direct, to heat a saturated solution in which is suspended an excess of the salt, as high as 177°. Doubtless, in this case the heating effect is aided by the heat of solution of the zinc chloride.

The theoretical explanation of the results of the experiments above described is comparatively simple. When water is heated the pressure exerted by its vapour increases until, on the attainment of boiling-point, it equals that of the atmosphere. The boiling-point of any solution under ordinary atmospheric conditions, is that temperature at which its vapour pressure is equal to the pressure of the atmosphere. The effect of the solution in water of any substance such as a salt is to reduce the vapour pressure of the liquid and hence to raise the temperature to which it must be heated before it will boil. It thus comes about that saline solutions have boiling-points higher than that of water. Now, when steam at the boiling-point of water is passed into a saline solution, the vapour pressure of the steam being equal to that of the atmosphere, but greater than that of the solution, the steam condenses in the solution and continues to do so as long as the latter has a lower vapour pressure—or, in other words, so long as the solution is not at boiling-point. When this point is reached the steam merely blows through the liquid, a sufficient quantity condensing to replace the heat lost by radiation, &c. It is to the liberation of the latent heat of the steam on condensation that is due the heating effect which we have been considering. When we reflect how great is the amount of this latent heat (some 537 calories for each c.c. of water condensed from steam) we can readily understand why such striking thermal effects are produced.

The fact of condensation taking place causes dilution of the solution with consequent reduction of boiling-point, unless a supply of the solid salt is maintained so as to keep the solution constantly saturated.

Taking into consideration all the facts, in my opinion the temperature to which a solution is heated by steam is a truer measure of its boiling-point than that obtained by the application of direct heat. It is well known that the temperature of boiling water varies within quite considerable limits, according to the nature of the vessel in which it is contained, and the boiling-point of liquids other than solutions of solids, is always determined from the temperature of the vapour. It is equally well known that saline solutions are very readily superheated when boiled over a flame, and it is highly probable that the boiling-point determined in this way will tend to be too high.

When alcohol of sp. gr. 0·816 is placed in the tube and steam injected, condensation takes place until the liquid becomes heated to boiling, and when this point is reached the liquid has a temperature of 79°1, that of the escaping vapour being 78°1. This difference is due to the fact that the vapour is richer in alcohol than the liquid, and accordingly, having a higher vapour pressure, comes away at a lower temperature. The fact that the water vapour escaping from a boiling saline solution has the same temperature as that from boiling water is due to the same cause.

While experimenting on heating saline solutions with steam, it is interesting to observe the effect on the thermometer placed in the vapour space of the boiling-tube when, through splashing, its bulb becomes covered with the saline solution. When a saturated solution of a moderately soluble salt is under observation and a portion of the liquid carrying particles of the solid salt splashes on to the thermometer bulb, the temperature at once rises to and remains at the same point as the boiling liquid itself. The film of saturated solution on the bulb, being enveloped in steam at 99° , behaves precisely like the mass of solution and maintains the thermometer at the boiling-point of the solution. Sodium chloride shows this extremely well.

The condensation of water from the atmosphere on particles of substances like sugar or salt, when the air is laden with moisture, is due to the same cause, the difference in vapour pressure of the film of solution adhering to the salt, and that of the moisture in the atmosphere, transference of water vapour always taking place in the direction of least vapour pressure. This was originally termed "invaporation" by Graham, who experimented with saline solutions in air saturated with water vapour, and determined the rate of condensation in the solutions (*Edinburgh Journ. of Science*, 1828, xvi., 249; *Report British Association*, 1886, 207).

In the course of some of my earlier experiments I used a vessel of boiling water in which to place the tube containing the solution under observation, but I quickly found that, far from being as efficient as the cotton-wool lagging subsequently used, the boiling water actually cooled the tube.

A knowledge of this property of steam whereby solutions are heated to temperatures considerably above that of the steam itself, may be of practical importance in chemical industries where saline solutions require heating to temperatures greater than 100° . Again, in some cases—as, for instance, in the course of operations in the manipulation of sugar and other organic substances—considerable over-heating may readily occur through the mistaken idea that the temperature of the steam used determines the limit of heating possible.

DISCUSSION.

Mr. W. A. DIXON said he had found that a steam spray immediately destroyed the prickly pear; was this partly due to the temperature being raised?

Dr. R. GREIG SMITH thought this matter had been explained in a clear and forcible way, and inquired what the temperature effect of direct steam on agar would be. This substance was used for bacteriological purposes, and could only be obtained as a solution after long and tedious digestion. Would direct steam hasten the process?

Mr. T. STEEL, in reply, said the efficiency of steam in killing prickly pear would be due to liberation of latent heat and thorough distribution; but unless some of the steam diffused into the sap, the temperature would not rise above that of the steam itself. As regards the effect of direct steam on agar-agar, he had no doubt that this substance would behave in the same manner as a salt.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances de l'Académie des Sciences. Vol. cxli., No. 26, December 26, 1905.

Researches on the Insoluble Potassium Compounds contained in Wood Charcoal.—M. Berthelot.—The action of dilute hydrochloric acid either hot or cold upon wood charcoal is to decompose the potassium and lime

salts of a certain insoluble acid, which latter is comparable with the acid produced during the action of the same concentrated hydracid on sugar. An investigation on this subject proves that in wood charcoal there exist two orders of alkaline and acid compounds which are equally stable. One series are immediately destroyed by dilute hydrochloric acid, the other being unattacked by this agent. Their properties and different affinities are analogous to those of the various classes of artificial and natural silicates, which behave so very differently towards acids.

Samarium Sulphates.—Camille Matignon.—Samarium sulphate can under suitable conditions be transformed either into an acid or basic salt. The anhydrous sulphate when added to excess of sulphuric acid and evaporated at about 200° leaves a residue of fine needle-shaped crystals of the basic salt, $\text{Sm}(\text{SO}_4\text{H})$. If this salt is calcined it is transformed into the neutral salt. When this neutral salt is heated to about 1000° it is transformed into the basic salt, $\text{Sm}_2\text{O}_3\text{SO}_3$. This latter is an amorphous powder very slightly yellow, and insoluble in water or cold dilute sulphuric acid.

Nickel Oxide.—H. Baubigny.—The author's series of experiments are to prove that MM. Bellucci and Clavari's theory of the non-existence of the oxides of nickel, Ni_2O_3 and Ni_3O_4 , is wrong, and that these oxides can be prepared.

Action of Acetylene on Anhydrous Iodic Acid.—George F. Jaubert.—The author investigates the action of acetylene gas on anhydrous iodic acid by using a U-tube containing some grammes of the anhydrous iodic acid heated to 80° in a water-bath, and examining the acetylene after this gas has been drawn through the tube by means of an aspirator. He finds that the substances act energetically on one another, and the iodic acid is reduced according to the equation $\text{I}_2\text{O}_5 + \text{C}_2\text{H}_2 = \text{I}_2 + 2\text{CO}_2 + \text{H}_2\text{O}$.

Action of Glucose on Selenious Acid.—MM. Œchsner de Coninck and Chauvenet.—During the process of reduction of selenious acid by means of glucose a variety of amorphous red selenium is produced which is insoluble in pure carbon disulphide. This appears to be in a physical condition corresponding to a colloidal state. It can be transformed partially into black selenium at about 100° .

Action of Ammonia Gas on Tri-bromide and Tri-iodide of Phosphorus.—C. Hugot.—Yellow phosphorus amide, which is produced when ammonia acts on the tri-bromide and tri-iodide of phosphorus at a very low temperature, is not soluble in ammoniacal ammonium bromide. It can be isolated, and gives, when slowly decomposed, phosphorus imide. On the contrary, it is very soluble in ammoniacal ammonium iodide. Its decomposition when contained in this liquid is effected slowly, and causes the precipitation of the less soluble imide.

A New Method of Preparation of Barium.—M. Guntz.—Some years ago the author showed that a metal containing 98.5 per cent of barium could be obtained by the calcination of barium amalgam in a vacuum. In his present research he varies the condition of this preparation without arriving at a very much more satisfactory state of purity. However, the dissociation of pure barium hydride in a vacuum at about 1200° allows of the preparation of the chemically pure metal.

Some New Derivatives of Pentabasic Phosphoric Acid, $\text{P}(\text{OH})_5$.—P. Lemoult.—The author finds that corresponding to the first series of etherobasic compounds of the form $\text{R}'-\text{O}-\text{P}(\text{NHR})_4$ is a second series of compounds $\text{R}''-\text{CO}_2-\text{P}(\text{NHR})_4$, which is much larger than the first one. The terms $\text{R} = \text{C}_6\text{H}_5$ have only been obtained in the second case.

Syntheses in the Series of Symmetric Heptanetriols 1.4.7.—J. L. Hamonet.—The author showed in a previous research that the bromo- or iodo-ether oxides $\text{RO}(\text{CH}_2)_n\text{X}$ give magnesium derivatives $\text{RO}(\text{CH}_2)_n\text{MgX}$, if n is greater than 2. He also showed the part which these substances play in various syntheses, particularly in

passing from a normal biprimary glycol to its superior homologue. He now describes a new application of these magnesium derivatives to a series of syntheses in the symmetric heptanetriols 1.4.7.

Derivatives from the Hydrogenation of Carvacrol.—Leon Brunel.—The author's experiments show that at a temperature of 115–120° catalytic hydrogenation is very active. Carvacrol is entirely transformed into hexahydroaromatic alcohol if the time of contact between the mixture of carvacrol, hydrogen, and nickel is sufficiently prolonged. On the other hand, the different results obtained at varying temperatures show that at 160° a similar secondary reaction giving rise to a second alcohol takes place. This action consists in the transitory formation of the acetone corresponding to a carvacromenthol called carvacromenthone, $C_{10}H_{18}O$. Above 150° traces of this acetone are formed by catalytic dehydrogenation of a part of the carvacromenthol which is produced.

MISCELLANEOUS.

Royal Institution.—On Thursday next, February 1, at 5 o'clock, Mr. Benjamin Kidd begins a course of two lectures at the Royal Institution on "The Significance of the Future in the Theory of Evolution"; and on Saturday, February 3, at three o'clock, Mr. J. W. Gordon delivers the first of two lectures on "Advances in Microscopy." The Friday Evening Discourse on February 2 will be delivered by Prof. S. P. Thompson on "The Electric Production of Nitrates from the Atmosphere," and on February 9 by Mr. H. F. Newall on "Eclipse Problems and Observations."

Institute of Chemistry.—Pass List of the January Examinations.—Of fourteen Candidates who entered for the Intermediate Examination, ten passed:—A. P. Davson; F. W. Foreman; W. Garsed; T. R. Hodgson, B.A. (Cantab.); T. J. Kirkland; A. Lathwood, B.Sc. (Lond.); B. D. W. Luff; J. F. Reid; H. Stanley, B.Sc. (Lond.); and F. Tattersfield. In the Final Examination for the Associateship (A.I.C.), of five examined in the branch of Mineral Chemistry, two passed:—E. R. Bullock, Assoc.R.C.Sc. (Lond.); and T. F. Cowie. Of four in Metallurgical Chemistry, two passed:—H. J. B. Rawlins, B.Sc. (Lond.); and Thomas Stenhouse, B.Sc. (Lond.), Assoc.R.C.Sc. (Lond.), A.R.S.M. Of four in Organic Chemistry, three passed:—W. P. Hayworth; F. H. G. Horsman, B.Sc. (Lond.); D. Spence, Ph.D. (Jena). Of twelve who entered in the branch of the Analysis of Food and Drugs, and of Water, including an Examination in Therapeutics, Pharmacology, and Microscopy, the following nine passed:—J. T. Cart, B.Sc. (Lond.); C. G. Gates, B.Sc. (Lond.); A. G. Holborow; Miss E. S. Hooper, B.Sc. (Lond.); S. J. Lewis, B.Sc. (Lond.); A. J. C. Lickorish; S. G. Liversedge; Miss E. A. Macadam; F. E. Thompson, Assoc.R.C.Sc. (Lond.). The Examiners in Chemistry were Mr. W. W. Fisher, M.A. (Oxon.), F.I.C., and Dr. G. G. Henderson, M.A., F.I.C. The Examination in Therapeutics, Pharmacology, and Microscopy was conducted by Dr. F. Gowland Hopkins, M.A., F.R.S., F.I.C.

MEETINGS FOR THE WEEK.

MONDAY, 29th.—Society of Arts, 8. (Cantor Lectures). "Modern Warships," by Sir William White, F.R.S., &c.

TUESDAY, 30th.—Royal Institution, 5. "Impressions of Travel in China and the Far East," by Prof. Edward H. Parker, M.A.

— Society of Arts, 8. "The Chemistry of the Painter's Palette," by Prof. J. M. Thompson, F.R.S., &c.

WEDNESDAY, 31st.—Society of Arts, 8. "The Garden City and the Cheap Cottage," by Thomas Adams.

THURSDAY, Feb. 1st.—Royal Institution, 5. "The Significance of the Future in the Theory of Evolution," by Benjamin Kidd.

— Society of Arts, 8. (Howard Lectures). "High Speed Electrical Machinery, with special reference to Steam Turbine Machines," by Prof. Silvanus Thompson, F.R.S.

— Chemical, 8.30. "Hydroxylamine- α - β -disulphonates (Structural Isomerides of Hydroxylamino-sulphates or Hydroxylamine- β -disulphonates)," by T. Haga. "Studies in the Camphane Series—Part XXI. Benzene-diazo- ψ -semicarbazinocamphor and its Derivatives," by M. O. Forster. "Relation between Absorption Spectra and Chemical Constitution—Part I. Chemical Reactivity of the Carbonyl Group; Part II. The Quinones and α -Diketones; Part III. The Nitranilines and the Nitrophenols," by E. C. C. Baly and A. W. Stewart. "Action of Light on Benzylidenephénylhydrazine," by F. D. Chattaway. "Union of Chlorine and Hydrogen," by D. L. Chapman and C. H. Burgess. "Molecular Weight of Adrenaline," G. Barger and A. J. Ewins. "Critical Temperature and Value of M.L./ θ of some Carbon Compounds," by J. Campbell Brown.

FRIDAY, 2nd.—Royal Institution, 9. "Electric Production of Nitrates from the Atmosphere," by Prof. S. P. Thompson, F.R.S., &c.

SATURDAY, 3rd.—Royal Institution, 3. "Advances in Microscopy," by John W. Gordon.

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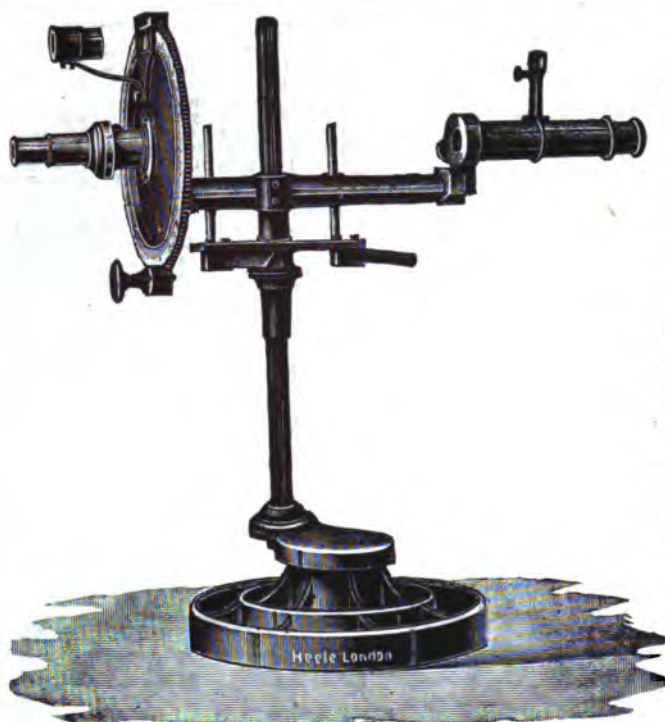
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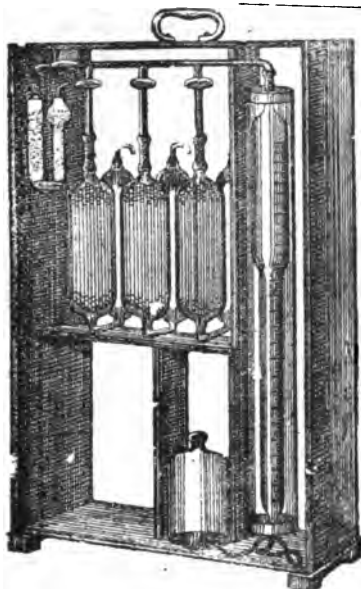
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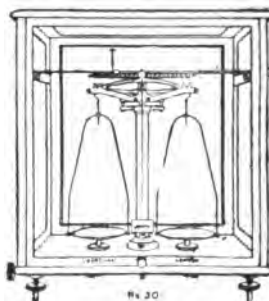
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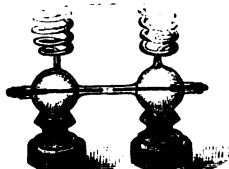
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THE CHEMICAL NEWS.

VOL. XCIII., No. 2410.

ON ANTI-FRICTION METALS HAVING LEAD AND TIN BASES.

By SERGIUS KERN, M.E., St. Petersburg.

THE practice of one of the principal engine-works in St. Petersburg showed that a babbitt, A, containing on the average 72 per cent of lead, 21 per cent of tin, and 7 per cent of antimony, used for three years in marine engines for lining heavy bearings, was especially good for the purposes intended.

The melting-point of this babbitt is 255° C., the freezing-point 242° C., leaving thus 13° for liquation effects. The last cypher represents an advantage of babbitt A over babbitts B and C, formerly used by the works, which have higher melting-points, but liquate more freely.

Babbitt B contains on the average:—Lead, 70 per cent; tin, 15 per cent; antimony, 15 per cent; melting-point, 260° C.; freezing-point, 233° C.; for liquation effects, 27°.

Babbitt C contains on the average:—Lead, 77 per cent; antimony, 22 per cent; melting-point, 260° C.; freezing-point, 237° C.; for liquation effects, 23°.

All these babbitts have lead as a base, and so far as my experiments show me, such babbitts liquate more freely in comparison with anti-friction metals having tin as a base.

In casting ingots of anti-friction metals we may clearly see, after analysing them, that such metals having a lead base give a more wide deviation in the percentage of elements entering into their composition, in comparison with metals having a tin base.

A certain moderate amount of lead is important in anti-friction metals with a tin base. It acts as "grease," and as a blinding element between tin and antimony.

To show the preference of tin base babbitts, we give some particulars of an anti-friction metal for bearings, of which we prepared large quantities:—The metal contained on the average—Tin, 84 per cent; antimony, 2.15 per cent; lead, 6.25 per cent; and copper 7 per cent. This babbitt melts at 225° C. and freezes at 221° C., leaving thus very little for liquation effects (4°).

NOTE ON THE PRODUCTION OF OZONE BY ELECTROLYSIS OF ALKALI FLUORIDES.*

By E. B. R. PRIDEAUX, M.A., B.Sc.

WHEN a concentrated aqueous solution of hydrofluoric acid or of a fluoride is electrolysed, it is reasonable to suppose that the products of the action of nascent fluorine on the water will be ozone, hydrogen peroxide, or a hypofluorite.

With regard, first, to the formation of hydrogen peroxide, Skirrow found that none was formed on electrolysing a mixture of hydrofluoric and sulphuric acids (*Zeit. Anorg. Chem.* 1902, xxxiii., 25–30). Pauli states that no hydrogen peroxide can be detected after the electrolysis of an alkaline solution of potassium fluoride (*Zeit. Electrochem.* 1897, iii., 474–498). I have confirmed Pauli's result and also his statement that no hypofluorite is formed. As he does not mention how he tested for the hypofluorite, I may be pardoned for saying that the method employed by me was to add potassium iodide to both neutral and alkaline solutions after prolonged electrolysis

under varying conditions. Certainly some free iodine can always be estimated by means of N/100 sodium thio-sulphate, but in every case a blank experiment carried out under the same conditions gave practically similar numbers.

The production of ozone from aqueous solutions of KF and of HF might, conceivably, have some commercial importance. There are two memoirs bearing on this point. Pauli (*loc. cit.*) states that in the electrolysis of neutral and alkaline solutions of potassium fluoride ozone is formed in considerable quantities. His evidence for this, however, consisted only in the powerful smell and attack of his rubber connections. Now it is known that an extremely small percentage of ozone will give quite a strong smell, and those who have worked with ozone know to their cost that very little will instantly destroy rubber connections. It seemed desirable, therefore, to analyse the anode gas and see how much ozone could actually be obtained in this way.

Gräfenberg (*Zeit. Anorg. Chem.*, 1903, xxxvi., [3], 355–380) electrolysed aqueous hydrofluoric acid of the usual commercial strength, collecting the anode gas under a platinum hood and passing it up into a bulb full of potassium iodide solution. By varying the current density he obtained percentages of ozone varying from 0.475 to 3.48 by volume. Now 3.48 is a very high percentage of ozone. By the silent discharge method it is reckoned that a gas may be obtained containing 0.5 to 5 per cent of ozone, while the highest recorded percentages I have been able to find in the literature are 14.39 (Moissan, "Le Fluor et les Composés," p. 131, by the decomposition of water by pure fluorine), and 17.5 (McLeod, *C. R.*, xlix., 591, by the electrolysis of dilute sulphuric acid under carefully regulated conditions). Experiments were therefore tried to see if as much as 3 per cent of ozone could easily be obtained from aqueous hydrofluoric acid. I did not find that this was the case, the percentage of ozone using a current density of 100 ampères per (d.cm.)² being 0.23.

The electrolysis of potassium fluoride is more promising commercially than that of hydrofluoric acid, since potassium fluoride solutions are both easier to deal with and do not lose strength so rapidly by evaporation and attack of the containing vessel as solutions of hydrofluoric acid.

In the first experiments the potassium fluoride solution was contained in a U-tube of thin glass coated inside with a mixture of paraffin and vaseline. The kathode was of copper, the anode of platinum foil. The hydrogen and oxygen were at first collected to make sure of the yield of gas, afterwards the percentages of ozone were calculated from the number of coulombs which had passed. A tandem bubbler of potassium iodide solution was used to estimate the ozone. It was found afterwards that the ozone in no case got through the first bubbler; the second one was therefore dispensed with. The U-tube was immersed in ice-water. Varying the current density between 5 and 10 ampères per square d.cm. made scarcely any difference to the yield, which was, however, considerably affected by the duration of the experiment, falling off as this increased. The largest yield was 0.65 per cent of ozone in twenty minutes, the smallest 0.096 in four hours twenty minutes. The current density in both these experiments was 10 ampères per d.cm. Variation of current density from 5 to 10 ampères (ten experiments) made little difference; increasing it to 120 ampères, however, much increased the percentage of ozone, which now, with half an hour's run, was as low as 0.1 per cent. It thus appears that the yield of ozone from saturated potassium fluoride solutions at 0° is in all cases quite low, certainly not exceeding 1 per cent.

Lewis's Quarterly List (No. 24, October, November, December, 1905).—This number contains upwards of 150 titles of books, a considerable number of which would be of special interest to readers of the CHEMICAL NEWS.

* A Paper read before the Faraday Society, January 30, 1906.

THE NATURE OF ENZYME ACTION.*

By E. FRANKLAND ARMSTRONG, Ph.D., D.Sc., A.C.G.I.

ENZYMES are of the greatest importance biologically, as being the agents by means of which very many of the changes—both destructive and constructive—which take place in animals and in plants are brought about.

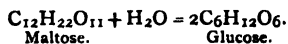
To the brewer, in particular, they have a wide significance, since they play a part in every stage of his daily work. It will, therefore, be at once granted, that for the proper scientific regulation of the technical process, it is essential to get as clear an idea of the nature of their action as possible.

When endeavouring to define an "Enzyme" the difficulty at once arises that no enzyme is as yet known in a pure state, or anything approaching it. In fact, all attempts to purify enzymes result sooner or later in the total loss of their activity; so that, even in the case of O'Sullivan's well-known investigations on the nature of invertase, there is no positive proof that the carbohydrate-like substances he isolated have anything to do with the specific activity of the enzyme. All that can at present be said is, that most enzymes can be obtained in an active state as white amorphous powders, soluble in water and containing carbon, hydrogen, oxygen, and nitrogen.

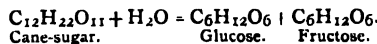
Generally speaking, they are active in presenting one or more molecules of water to complex substances and breaking them down into simpler compounds. They are non-living, inasmuch as they cannot increase by reproduction, differing in this respect from the yeasts, moulds, and bacteria. The so-called vital changes brought about by these latter agents are, however, now generally regarded as due to the enzymes they contain, and which they have in some way elaborated from their protoplasm.

Enzyme extracts or solutions of enzymes in water are conveniently prepared by grinding or shaking the particular animal or vegetable matter used with water or dilute alcohol in presence of an antiseptic to exclude bacterial contamination, toluene being by far the best. The active principle can be precipitated from such an extract by cautious addition of alcohol, though this procedure diminishes the activity. It is often necessary to rupture the cell wall by first drying the material and powdering it with glass or Kieselguhr before extraction, as the enzyme cannot diffuse through the undamaged cell wall.

Turning to the consideration of a few specific examples, it is found that the extract made by grinding up malt with water contains an enzyme—*diastase*—which converts starch into unfermentable dextrins and maltose, but is without further action on these products. Pressed yeast, dried by spreading it in thin layers on unglazed biscuit ware, when extracted with water yields an enzyme—*maltase*—which will convert a molecule of maltose into two molecules of glucose—



The dried yeast extract is also able to invert or hydrolyse cane-sugar, converting it into a molecule of glucose (dextrose) and a molecule of fructose (lævulose)—



This change is brought about by an enzyme—*invertase*—the term inversion having been originally applied to the change to denote the change in the optical rotatory power of the solution as the cane-sugar, which has a positive rotatory power, is converted into the mixture of the two sugars having a negative rotatory power.

It would seem therefore that yeast extract contains two different enzymes: it may be well at this point to consider

briefly the evidence for believing that the two enzymes are really different.

(a) If a yeast extract be precipitated with alcohol, and the precipitate be re-dissolved in water and the process be repeated once or twice, an extract is obtained which, though it is still capable of hydrolysing cane-sugar, is quite without action on maltose. Similarly, an extract, prepared by allowing yeast to stand for a few days until it becomes liquid, then precipitating the enzyme carefully by means of alcohol and dissolving this precipitate in water, will attack cane-sugar only, and not maltose.

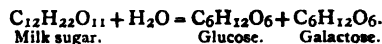
(b) A cold water yeast extract when used at high temperatures—above 40° C.—loses its power of hydrolysing maltose. Thus the power of the yeast extract to hydrolyse cane-sugar can be retained when its action on maltose has ceased.

(c) Further, such peculiar races of yeast as *Saccharomyces Marxianus* and *Saccharomycodes Ludwigii* when dried furnish extracts which do not contain any maltase, though still able to invert cane-sugar and therefore containing invertase.

(d) Whereas, yeasts such as *Schizosaccharomyces octosporus* and *Saccharomycopsis capsularis* (extracts from which hydrolyse maltose with ease) are entirely without action on cane-sugar, i.e., they contain no invertase.

It is thus possible to obtain yeast extracts which sometimes contain invertase and no maltase, sometimes maltase alone, sometimes both enzymes. No clearer proof that the two enzymes in question are distinct substances can be desired.

As a further example the enzyme *lactase* may be cited. This converts milk sugar (lactose), an isomeride of cane-sugar and maltose, into two sugars, glucose and galactose:—



It occurs in the so-called Kefir grains and also in some rare varieties of yeasts such as *Saccharomyces fragilis*. Neither invertase nor maltase has any action on milk sugar, whilst the lactase extract does not hydrolyse maltose.

There is thus every proof that lactase, maltase, and invertase are distinct enzymes.

The biase sugars, maltose and cane-sugar, just dealt with, are known to the brewer as fermentable sugars. It is important to be quite clear, however, that in reality fermentation can only take place after the biase sugar, through the agency of the appropriate enzyme, has been converted into the simpler C₆ sugars. Hydrolysis must precede fermentation: the only directly fermentable sugars are glucose, fructose, galactose, and a somewhat rarer sugar—mannose—all of which have the same composition, C₆H₁₂O₆. In proof of this, the fact may be advanced that although *octosporus* yeast ferments glucose and fructose easily, it is quite without action on cane-sugar. When, however, a single drop of an invertase extract is added a brisk fermentation sets in, proving that inversion of the cane-sugar is necessary before fermentation can take place. In like manner, *Marxianus*, which ferments glucose strongly, cannot ferment maltose until used along with a maltase extract. The addition of an invertase extract containing no maltase has no effect. Similarly, it is impossible to ferment milk sugar by any yeast which does not contain lactase, unless a lactase extract be also used first to hydrolyse the biase.

The foregoing illustrations afford definite proof, not only that the various enzymes mentioned differ from one another, but that a different specific enzyme is required to hydrolyse each material. The action of enzymes may be said to be *selective*, and to differ therefore from the action of acids. Any one acid—such as hydrochloric acid—can hydrolyse any and all of the biase sugars or starch or similar compounds, though, it is true, with different degrees of readiness, whereas it has been shown that invertase acts on cane-sugar only.

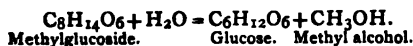
* From the *Journal of the Institute of Brewing*, xi., No. 6, July—ept., 1905.

Having established that maltose is affected only by maltase, it becomes of interest to ascertain whether any other substances can be found which maltase will act upon, and, if so, to determine the relationship that such compounds bear to maltose. It was not until certain developments which could be utilised had taken place in synthetical chemistry that this step forward could be realised.

By the interaction of glucose and methyl alcohol in presence of hydrogen chloride, a substance— $C_7H_{14}O_6$ —termed methylglucoside, is formed; whilst at the same time, as is so often the case in chemical reactions, an isomeride (that is a substance possessing the same constitution but differing in physical properties) is obtained.

These two isomeric methylglucosides are distinguished by the prefixes α - and β -.

Maltase readily converts the α -methylglucoside into glucose and methyl alcohol:—

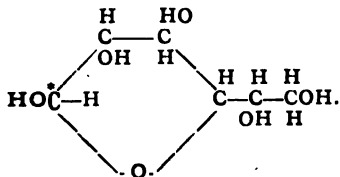


but is totally without action on the β -isomeride. The enzyme can therefore discriminate between the two isomerides. In order to realise the full significance of this fact a few words must be devoted to the structure of these compounds.

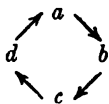
The β -methylglucoside, though not affected by maltase, can be hydrolysed by means of *emulsin*, an enzyme obtained by macerating sweet almonds with water. *Emulsin* also hydrolyses amygdalin and many of the natural glucosides, but it is totally without action on either α -methylglucoside or maltose.

Thus *emulsin* or maltase afford a ready means of distinguishing α - or β -methylglucosides.

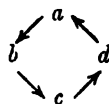
The behaviour of glucose, $C_6H_{12}O_6$, the parent substance of the glucosides, is best denoted by the following structural formula,* which I have elsewhere given reasons for (*Trans. Chem. Soc.*, 1903, 1310; 1904, 1044).



The methylglucosides are derived from this by substituting an OCH_3 group for the OH group attached to the carbon atom marked*. This carbon atom is what is known as asymmetric, in that it has four different groups (a, b, c, d) attached to it. These groups can obviously be written in order either clockwise—



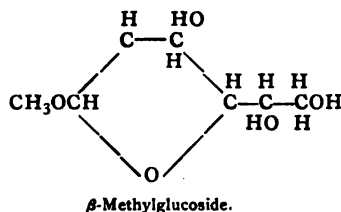
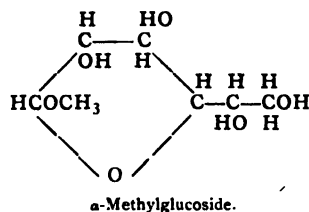
or counter-clockwise—



Two different forms are therefore possible, related as object to image, and they are termed stereoisomerides.

The isomeric methylglucosides are related in this manner; the difference between them may be represented by writing the OCH_3 group either to the right or to the left

of the carbon chain and the H on the other side. We do not at present know which of these represents the α - and which the β -form, but it is convenient here to label them—



The properties of the two glucosides are compared in the following table:—

	α -Methylglucoside.	β -Methylglucoside.
Melting-point	165°	104°
Optical rotatory power . . .	+157°	-33°
Behaviour towards maltase . .	Hydrolysed.	—
Behaviour towards <i>emulsin</i> . .	—	Hydrolysed.

The action of the enzyme on these glucosides is to eliminate the OCH_3 and replace it by OH. It appears that the fact that this OCH_3 is sometimes on the one side of the molecule and sometimes on the other is enough to render the hydrolysis impossible. It may well be supposed that the maltose is attracted to the α -glucoside molecule, as pictured above, on its right-hand side, and can then hydrolyse the α -glucoside: that it is incapable, however, when so attached of acting on the OCH_3 group should this be in the β -position on the other side of the molecule. Similarly, perhaps, *emulsin* is attached to the left-hand side of the molecule and can then deal with the OCH_3 group in its left-hand or β -position, but it is quite out of harmony with this group in its right-hand or α -position.

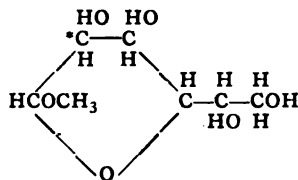
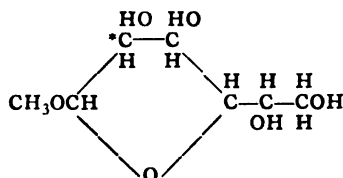
Such a simple conception as the above affords a complete explanation of the differential attack of the α - and β -glucosides; it involves, however, one assumption of fundamental importance, namely, that the enzyme becomes attached to the glucoside it hydrolyses.

A large number of experimental facts in favour of this view have been brought forward. It will here suffice to point out the very close connection between molecular structure or configuration, as it is termed, and liability to attack by enzymes.

Referring to the structural formula for glucose, it will be seen that each carbon atom has an OH and H radicle attached to it. Just as interchange of the position of the OCH_3 and H radicles on the asymmetric carbon was shown to produce two isomerides, so interchange of the OH and H on any other of the asymmetric carbon atoms in glucose will produce a new isomeride. In all sixteen isomerides of glucose related in this manner are possible, and no less than thirty-two isomeric glucosides. Relatively few of these compounds occur naturally; a number have been synthetically prepared by Emil Fischer, so that no less than eleven of the possible sixteen isomerides of glucose and many of the glucosides are known.

The two methylmannosides may be represented by the following formulæ:—

* Strictly speaking, glucose in solution is a mixture of two isomerides having the same relationship as the α - and β -glucosides,

 α -Methylmannoside. β -Methylmannoside.

It will be seen that they differ from the α - and β -methylglucosides only in respect to the relative positions of the groups attached to the carbon atom marked * contiguous to that to which the OCH_3 is attached. This slight difference, however, is enough to make both mannosides absolutely resistant to the attack of either maltase or emulsin, and as yet no enzyme is known which will hydrolyse them.

In general, any other change in the configuration of the molecule is found by experiment to render the resulting glucoside incapable of being attacked by any known enzyme, and the same applies to compounds derived from sugars, either with one carbon atom less in the chain, as arabinose or xylose ($\text{C}_5\text{H}_{10}\text{O}_5$) or with a carbon atom more, such as glucoheptose ($\text{C}_7\text{H}_{14}\text{O}_7$).

Lastly, the methylglucosides derived from *l*-glucose, the mirror-image of ordinary or *d*-glucose, both resist the action of either maltase or emulsin.

These illustrations will suffice to show the very intimate relationship which exists between an enzyme and the substance it acts upon: this can only be explained by supposing some form of combination between the two. The enzyme, moreover, must fit the glucoside at every point along the chain of carbon atoms. The combination may perhaps be compared to the way in which the successive fingers of a glove fit on to a right hand; if the position of any one finger be altered, it is impossible to fit the glove; further, the glove will not fit on the left hand.

Further evidence of the formation of some form of compound between enzyme and carbohydrate has been obtained by studying the action of maltase on maltose in the presence of a large number of other substances. It was found that of all the substances tried only glucose and the glucosides caused a retardation in the rate of change, presumably because these combined with the enzyme and so prevented part of it from acting on the maltose. All the other substances tested, although in many cases they differed only slightly from glucose, produced no appreciable effect, showing that any alteration in structure, however small, was sufficient to prevent combination with the enzyme.

It is perhaps necessary to point out that the actual hydrolysis of the carbohydrate is due to the action of the water molecules. The enzyme may be conceived, perhaps, as acting as a vice in presenting in the appropriate manner the water molecule to the centre to be hydrolysed.

The facts here discussed can be very easily followed by means of a solid model in which the affinities of the carbon atom are represented as emanating from the centre of a regular tetrahedron. When such a model is used, the alterations in the surface presented, produced by interchanging the positions of H and OH groups, are very striking.

A short discussion followed the reading of the paper.

A STUDY OF ROCK DECOMPOSITION UNDER THE ACTION OF WATER.*

By ALLERTON S. CUSHMAN.

IN a previous publication the author has pointed out that when most rocks are crushed down to fine powders (100 mesh and finer) and then wetted, immediately hydrolytic reactions occur, which point indubitably to the fact that certain decompositions of the rock substance have taken place. Whenever, as is almost invariably the case in the rocks of igneous origin, alkaline bases, such as lime, magnesia, soda, and potash, are present, the fine powder will show an immediate alkaline reaction to delicate indicators, such as phenolphthalein, the instant the water comes in contact with them. Although these reactions start with remarkable promptness, they do not go far, and the actual amount of alkali set free is so small that few investigators have paid attention to the matter. Undoubtedly, however, as it is not difficult to show, these decompositions are of the very highest importance, not only in their relation to certain interesting theoretical considerations having to do with physical chemistry, but also because they have a direct bearing upon many important problems of engineering and of agricultural and industrial chemistry. The value of a rock for road building, so far as its binding or cementing power is concerned, depends entirely upon the fact that decomposition takes place. Since rocks, even of the same species and type, vary widely in their resistance to the action of water and dilute solutions when in the state of fine dust or powder, the value of such investigations as are detailed in this paper becomes apparent. Storer, from the standpoint of agricultural chemistry, discusses in a most interesting way the disintegrating power of water on rocks ("Agriculture in some of its Relations with Chemistry," 5th ed., vol. i., p. 134). Describing the work of the Messrs. Rogers, who found that from a third of 1 per cent to 1 per cent of some minerals was dissolved out by water, Storer calculates that, granting only one third of a pound of potash is dissolved out of every 100 pounds of rock, then in every 3,500,000 pounds which would go to make up an acre taken to the depth of 1 foot, about 10,000 pounds might be dissolved. Since 15 tons of good stable manure will supply no more to an acre than 150 pounds of potash alone, it is at once apparent that the immediate decomposition of certain potash-holding rocks under the action of water is a question of very high importance.

Daubree found that if felspar was ground in an iron cylinder for one hundred and ninety-two hours with water enough to make a thin slip, 0.4 per cent of potash could be found in solution at the end of the run. This result is in substantial agreement with numerous similar experiments which have been made in this laboratory. In the experiments about to be described, a typical orthoclase felspar from Westchester County, N.Y., was selected and ground dry in a ball mill until the powder all passed a 100-mesh screen. This powder had the following composition:—

	Per cent.
SiO_2	66.23
Al_2O_3	19.96
Fe_2O_3	0.25
K_2O	11.61
Na_2O	1.39
P_2O_5	0.39
$\text{H}_2\text{O } 100^\circ +$	0.28
100.11	

If this orthoclase dust was directly extracted with distilled water by shaking 25 grms. with 100 c.c. of distilled water and the mixtures subsequently filtered, about 0.024 per cent of soluble alkali was found by titration in the

* United States Department of Agriculture, Office of Public Roads, LOGAN WALLER PAGE, Director.

clear filtrate. The author has, however, shown elsewhere that by no means all of the alkaline bases set free by the decomposing action of water pass freely into solution. (See *U.S. Dept. Agr., Bureau of Chemistry*, Bull. No. 92). By subsequent action of some electrolyte, such as NH_4Cl , more of the base is carried into solution. This is shown in Table I. A number of samples were weighed out in Jena glass flasks, varying amounts of NH_4Cl added, and the contents thoroughly shaken with a measured quantity of water. Aliquot portions of the filtrates were evaporated to dryness, the residue heated to expel all ammonia salts, and weighed.

TABLE I.—Orthoclase Rock Powder treated with Dilute Solutions NH_4Cl .

Amount taken.	Strength NH_4Cl solution (100 c.c. used).	Weight of residue.	Residue per 100 grms.
Grms.	Per cent.	Grm.	Grm.
25	0'000	0'0060	0'0240
25	0'001	0'0092	0'0368
25	0'010	0'0184	0'0736
25	0'100	0'0400	0'1600
25	1'000	0'0624	0'2496
25	1'500	0'0664	0'2656
25	2'000	0'0608	0'2432

Table II. is included here to show that rock powders other than orthoclase are subject to similar decompositions. (See *U.S. Dept. Agr., Bureau of Chemistry*, Bull. No. 92).

TABLE II.—Diabase Rock Powder treated with Dilute Solutions NH_4Cl .

Amount taken.	Strength NH_4Cl solution (100 c.c. used).	Weight of residue.	Residue per 100 grms.
Grms.	Per cent.	Grm.	Grm.
25	0'000	0'0064	0'0256
25	0'001	0'0092	0'0368
25	0'010	0'0180	0'0730
25	0'100	0'0616	0'2464
25	1'000	0'1412	0'5648

The results included in these tables show the amount of soluble alkali that can be obtained in solution from the mere digestion of ordinary dry ground rock powders in cold water and dilute solutions of an electrolyte. If the powders are ground wet in a mill for a number of hours, more decomposition takes place, and more free alkali passes into solution. This is clearly shown in Table III.

TABLE III.—Orthoclase Rock Powder treated with NH_4Cl after Wet Grinding.

Amount taken.	Strength NH_4Cl solution (100 c.c. used).	Weight of residue.	Residue per 100 grms.
Grms.	Per cent.	Grm.	Grm.
25	0'000	0'080	0'320
25	1'000	0'118	0'472
25	1'000	0'125	0'500
25	1'000	0'132	0'528
25	1'000	0'143	0'572

The action of dilute solutions of ammonium chloride in causing an increased amount of free alkali to pass into solution has been discussed in a previous publication. (See *U.S. Dept. Agr., Bureau of Chemistry*, Bull. No. 92). The work of Linder and Picton (*Journ. Chem. Soc.*, 1892, li., 114, 148), Whitney and Ober (*Journ. Am. Chem. Soc.*, 1901, xxiii., 842), and others has shown that when colloid or pectoid substances are formed in the presence of solutions of electrolytes, the bases generally will be absorbed by the pectoid substance, if the pectoid is electro-negative in character. Although these absorbed bases can not be removed by water alone, they can be removed either wholly or in part by digestion in dilute solutions of certain electrolytes. The writer has shown that when water acts upon certain minerals and rocks, decomposition immediately commences, leading to simultaneous formation of pectoid matter and soluble electrolytes. We should therefore expect only a small portion of the alkaline bases

set free to pass freely into solution, which is precisely what we find to be the case.

If water attacks a mineral powder like orthoclase so readily, why, we may ask ourselves, does the reaction, practically speaking, come to a definite end almost immediately? The answer to this question is simple. Any reaction in which the reaction products are insoluble in water and are not removed by any agency from the surface of solid particles where they are formed, would behave in exactly this way. Remove the reaction products from the immediate place of formation, either by stirring, grinding, or by the action of certain electrolytes, and the reaction will proceed.

In spite of the general soundness of this reasoning, it was felt that experimental evidence was needed in order to get light upon the extent to which these decompositions actually take place. The vexatious difficulties experienced in trying to filter slimy suspensions, the effect of carbonic acid upon the indicator used in volumetric work, and, above all, the uncertainty as to the extent to which the soluble bases actually pass into solution, make investigations of this kind extremely difficult. It is probable, however, that the results obtained point the way to, even if they do not measure the quantitative limits of the reactions under discussion. It is certain that the kaolinisation of feldspars in nature proceeds to the conclusion of the decomposition reactions, but, although the process may be observed in some places as at present going on, we have no data on the time necessary to complete it, nor on the exact conditions in nature most likely to accelerate it. It may be surmised that the decomposition is extremely slow, and that natural attrition under the action of water is not so very much unlike the wet grinding operations of a laboratory mill.

In attempting to measure quantitatively the extent of the decomposition reactions as observed in the laboratory, there was always the danger that a portion, if not all, of the free alkali obtained already existed as such in the orthoclase sample, and was not therefore the result of the immediate decomposition induced by the action of the water. After some experimentation, a method was hit upon which seems in every respect suitable for testing the points just raised, as well as supplying an excellent laboratory method for the separation and analysis of various sorts of slimes and pectoid substances, which for obvious reasons are difficult, if not impossible, to filter. In our experiments, definite weights of the rock powders are slimed with a measured quantity of water, and the slime placed inside an ordinary unglazed porcelain cup such as is used for certain common types of bell batteries. An ordinary arc-light carbon is now inserted and the porcelain cup set down inside a glass cylindrical vessel containing distilled water to the depth of 2 or 3 centimetres. Another carbon electrode is then inserted in the outside compartment. The cell is now connected with a direct current of about 110 volts, the inner or slime chamber being in connection with the positive pole.

Under the action of electrical endosmosis all the liquid from the anode or slime chamber passes into the cathode chamber, while at the same time, owing to electrolysis, the free alkali passes out in a condition in which it can be easily titrated or otherwise estimated. In the course of a few hours the slime has been transformed into a hard, comparatively dry cake, while the free alkali has been partially removed. The alkaline liquor in the cathode chamber is now poured out into a beaker and titrated with tenth normal acid. After re-sliming the material in the anode chamber with the proper quantity of water, the apparatus is started again. With the conditions under which we were working it was found that about one hour sufficed for each run, and a series of runs was made, the results of which are shown in Table IV.

The results are seen to better advantage when plotted; the amount of alkali extracted by each run falls off rapidly until it practically reaches zero. The amount of current passing was approximately about 0'05 of an ampere when

the voltage was about 120. Another sample of orthoclase from an entirely different locality gave a very similar result.

Since it is quite possible by this method to extract practically all the free alkali which is contained in the slime, it is apparent that a re-grinding of the orthoclase and re-electrolysis will prove whether or not the alkali existed originally free in the orthoclase sample. In order to put this point to the test, the orthoclase charge which had been electrolysed to the end of the yield of free alkali was re-ground for several hours in a porcelain pebble mill. It was then slimed again with water, and re-electrolysed. *The results are conclusive as to the fact that the alkali which is obtained is set free by the immediate action of water, and is not originally a free constituent of the rock powder.*

The writer has contended in a number of publications that when most rock powders are acted on by water, only a portion of the soluble alkaline bases set free pass into solution, owing to the absorption by the pectoids which are also products of the decomposition. A mass of evidence of various sorts has been collected to support this view. This has a wide practical bearing, as it explains why the larger proportions of the bases are conserved during the natural process of rock disintegration, instead of being leached out and washed away. The results given above furnish additional proof of the truth of this contention. The fact that the actual amount of alkali extracted by the second electrolysis is larger than that extracted by the first is probably due to the increased surface effect resulting from the finer grinding. The equilibrium between the absorbent effect of the pectoids and the solvent effect of water requires further investigation. Orthoclase powder simply leached with water as is shown in Table I, yielded only 0.024 per cent of soluble alkali, whereas by endosmosis

TABLE IV.—Electrolysis of Orthoclase.

No. 1242.

Length of runs. Mins.	N/10 HNO ₃ taken. C.c.	KOH equivalent. Grm.	KOH extracted per 100 grms. Grm.
<i>First Electrolysis.</i>			
60	11.10	0.06216	0.03108
60	10.20	0.05712	0.02856
60	7.80	0.04364	0.02182
60	6.60	0.03696	0.01848
60	3.90	0.02184	0.01092
60	3.30	0.01848	0.00924
60	3.70	0.02072	0.01036
60	2.70	0.01512	0.00756
60	2.40	0.01344	0.00672
60	2.00	0.01120	0.00560
60	1.40	0.00784	0.00392
60	1.00	0.00560	0.00280
60	1.20	0.00672	0.00336
60	0.75	0.00420	0.00210
60	0.60	0.00336	0.00168
60	0.50	0.00280	0.00140
60	0.35	0.00196	0.00098
60	0.35	0.00196	0.00098
60	0.50	0.00280	0.00140
60	0.40	0.00224	0.00112
60	0.05	0.00028	0.00014
60	0.25	0.00140	0.00070
(a)	0.30	0.00168	0.00084
75	0.80	0.00448	0.00224
60	0.20	0.00112	0.00056
60	0.15	0.00084	0.00042
75	0.20	0.00112	0.00056
70	0.10	0.00056	0.00028
63	0.05	0.00028	0.00014
60	0.05	0.00028	0.00014
Total ..	62.90	0.35220	0.17610

TABLE IV.—Electrolysis of Orthoclase (continued).

Length of runs. Mins.	N/10 HNO ₃ taken. C.c.	KOH equivalent. Grm.	KOH extracted per 100 grms. Grm.
<i>Second Electrolysis.</i>			
60	13.30	0.07448	0.04598
60	15.00	0.08400	0.05185
60	13.00	0.07280	0.04494
60	8.50	0.04760	0.02938
57	5.80	0.03248	0.02005
60	4.10	0.02296	0.01417
75	4.40	0.02464	0.01521
60	0.80	0.00448	0.00277
60	0.30	0.00168	0.00104
60	0.20	0.00112	0.00069
60	0.20	0.00112	0.00069
(b)	1.50	0.00840	0.00519
Total ..	67.10	0.37576	0.23196
<i>Third Electrolysis.</i>			
60	6.50	0.03640	0.02459
60	17.00	0.09520	0.06432
60	17.50	0.09800	0.06622
60	15.50	0.08680	0.05865
58	11.60	0.06496	0.04389
58	11.20	0.06272	0.04238
58	10.70	0.05992	0.04049
60	7.80	0.04368	0.02951
60	5.50	0.03080	0.02081
60	3.50	0.01960	0.01324
60	2.30	0.01288	0.00870
60	2.30	0.01288	0.00870
60	2.10	0.01176	0.00795
60	1.75	0.00980	0.00662
60	1.80	0.01008	0.00681
63	1.40	0.00784	0.00530
60	1.40	0.00784	0.00530
60	1.65	0.00924	0.00624
62	1.00	0.00560	0.00378
60	0.90	0.00504	0.00341
60	0.70	0.00392	0.00265
60	0.70	0.00392	0.00265
60	0.80	0.00448	0.00303
60	0.60	0.00336	0.00227
(b)	0.70	0.00392	0.00265
60	0.70	0.00392	0.00265
60	0.25	0.00140	0.00095
62	0.25	0.00140	0.00095
60	0.10	0.00056	0.00038
(b)	2.10	0.01176	0.00795
Total ..	130.30	0.72968	0.49304
No. 1300.			
<i>First Electrolysis.</i>			
60	14.00	0.07840	0.03920
60	10.50	0.05880	0.02940
60	6.10	0.03416	0.01708
60	5.10	0.02856	0.01428
60	4.40	0.02464	0.01232
60	4.40	0.02464	0.01232
60	2.80	0.01568	0.00784
60	2.70	0.01512	0.00756
60	1.80	0.01008	0.00504
60	1.50	0.00840	0.00420
58	1.10	0.00616	0.00308
60	1.60	0.00899	0.00449
60	0.70	0.00392	0.00196
60	0.60	0.00336	0.00168
57	0.50	0.00280	0.00140
60	0.35	0.00196	0.00098
60	0.20	0.00112	0.00056
Total ..	58.35	0.32679	0.16339

TABLE IV.—*Electrolysis of Orthoclase (continued).*

Length of runs. Mins.	N/10 HNO ₃ taken. C.c.	KOH equivalent. Grm.	KOH extracted per 100 grms. Grm.
<i>Second Electrolysis.</i>			
60	11'40	0'06384	0'03395
60	13'50	0'07560	0'04021
60	18'30	0'10248	0'05451
60	9'90	0'05544	0'02949
60	4'50	0'02520	0'01340
60	2'40	0'01344	0'00715
60	2'30	0'01288	0'00685
60	1'20	0'00672	0'00357
60	0'70	0'00392	0'00209
60	0'65	0'00364	0'00194
64	0'45	0'00252	0'00134
68	0'30	0'00168	0'00089
60	0'25	0'00140	0'00074
60	0'65	0'00364	0'00194
62	0'35	0'00196	0'00104
64	—	—	—
(a)	5'10	0'02856	0'01519
Total ..	71'95	0'40292	0'21430
<i>Third Electrolysis.</i>			
60	35'00	0'19600	0'11137
60	11'90	0'06664	0'03786
60	2'80	0'01568	0'00891
62	1'80	0'01008	0'00573
60	0'70	0'00392	0'00223
60	0'40	0'00224	0'00127
60	0'30	0'00168	0'00095
63	—	—	—
243	0'90	0'00504	0'00286
(b)	4'40	0'02464	0'01398
Total ..	58'20	0'32592	0'18516
(a) Over Sunday.			(b) Over night.

it was possible to obtain 0.16 per cent. It is impossible to avoid the conclusion that the difference of about 0.15 per cent represents alkali absorbed by the pectoid aluminum silicate which the microscope reveals existing as cloggy films upon and among the rock particles when the action has been accentuated by wet grinding. Furthermore, the quantity of free alkali extracted by the action of electrolysis and endosmosis agrees sufficiently well with the quantity that is extracted from wet orthoclase by treatment with ammonium chloride.

Bredig and Schwerin have lately called attention to the value of the application of the principles of electrical endosmosis to certain practical problems (*Zeit. Angew. Chem.*, 1903, xvi., 581). The phenomenon was first studied by Wiedemann in 1852 (*Pogg. Ann. Phys.*, 1852, lxxxvii., 350), and it was shown that the power with which a galvanic current will drive a given liquid through a porous diaphragm of given area Q in the direction of the negative pole depends directly upon the hydrostatic pressure developed in the difference of level h , the intensity of the current J , the specific resistance of the liquid m , the thickness of the porous wall d , and inversely upon the area of the diaphragm. From these facts the following formula was derived:—

$$P = h = k \frac{J.m.d}{Q}$$

The particular interest of this formula for the work in hand is that it seems to offer a possible method for studying quantitatively the relative amount of decomposition which takes place in various rock powders under the action of water.

Aside from its quantitative possibilities, electrical

endosmosis offers so efficient a method for separating not only water, but also acids or alkalis from slimy, colloidal, and otherwise unfilterable mixtures, that it is very surprising that it has not found a standard place both in the laboratory and in technical practice.

The question whether by this method, *i.e.*, successive grindings of orthoclase with removal of the alkaline decomposition products by electrical endosmosis, the kaolinisation of orthoclase and other rock powders can be carried on in the laboratory to the completion of the reaction as it undoubtedly is in nature, remains a field for future study. There would seem to be little doubt that it can be done. In any case, the present line of inquiry seems to offer an interesting and novel method for studying rock decomposition.

These investigations, of which this paper contains but a preliminary account, are being carried on, and will be presented in detail at some future time.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, January 18th, 1906.

Prof. R. MELDOLA, F.R.S., President, in the Chair.

MESSRS. J. E. Coates, A. Amos, and M. B. Jack were formally admitted Fellows of the Society.

The PRESIDENT announced that the Society had lost a distinguished Fellow in the person of Professor Hermann Johann Philipp Sprengel, Ph.D., F.R.S., who was elected in December, 1864, and died on January 14th, 1906.

It was also stated by the President that the Society was indebted to Sir Henry E. Roscoe, F.R.S., for an important donation to the Library of nearly one hundred alchemical and old chemical works of great interest and value.

Certificates were read for the first time in favour of Messrs. Richard Victor Briggs, Sirseah Research Station, Mozufferpore P.O., Bengal, India; Llewellyn Thomas Jones, B.Sc., Brenig View, Tregaron; Samuel Parfitt, 33, Partridge Road, Cardiff; Robert Le Rossignol, B.Sc., 2, Cæsarea Place, Jersey; Arthur William Thorp, 210, Sherborne Road, Yeovil; Henry Edgar Watt, B.Sc., 19, Summerhill Road, Dartford, Kent; Richard Willstätter, Ph.D., Bergstrasse 25, Zürich V.

Certificates have been authorised by the Council for presentation for ballot under By-law I. (3) in favour of William Mace, of Hope, Kingston, Jamaica; George Stanley Talbot, Ocean Island, Sydney, N.S.W.; Harry Sands Grindley, D.Sc., State University, Illinois, U.S.A.

The Council has ordered the following letter and report to be printed in the *Journal* and *Proceedings* of the Society:—

Government Laboratory,
Clement's Inn Passage, Strand, London, W.C.,
November 10th, 1905.

GENTLEMEN,—I have the honour to transmit to you the Report of the International Committee on Atomic Weights, 1906, to which I have appended the signatures of Professors Moissan and Seubert as desired by them.

It will be seen that the Committee, for the reasons stated in the report, are not prepared to advise any immediate alteration in the values for the atomic weights given in their last report. They recommend, however, in conformity with the desires of the great majority of the Larger Committee, that the values in the table for 1906 be given on the basis of $O = 16$, to the exclusion of those on the basis of $H = 1$.—I am, Gentlemen, your obedient Servant,

T. E. THORPE.

The Hon. Secretaries, the Chemical Society,
Burlington House, London, W.

Report of the International Committee on Atomic Weights.

During the year 1905 there has been unusual activity in the determination of atomic weights, and some of the work done relates to the most fundamental values. The entire system of atomic weights is thus affected more or less profoundly, and a general revision of the table would seem to be needed within the near future. Neglecting minor investigations, the more important determinations published since our last report are briefly as follows:—

Cadmium.—Atomic weight determined by Baxter and Hines (*Journ. Am. Chem. Soc.*, 1905, xxvii., 222), by analysis of the chloride. Three measurements of the ratio $\text{CdCl}_2 : 2\text{AgCl}$ gave in mean $\text{Cd} = 112.476$. Six measurements of the ratio $\text{CdCl}_2 : 2\text{Ag}$ gave in mean $\text{Cd} = 112.462$. General mean of both series, 112.469; when $\text{Ag} = 107.93$ and $\text{Cl} = 35.473$. As additional determinations are promised, based upon a study of cadmium bromide, a change in the atomic weight of cadmium as given in our table may properly be deferred.

Carbon.—From the ratios published in 1904, relative to the basic acetate and acetylacetate of glucinum, Parsons (*Journ. Am. Chem. Soc.*, 1905; xxvii., 1204; *Zeit. Anorg. Chem.*, 1905, xlv., 215) has computed the atomic weight of carbon. The values obtained by treating the two ratios algebraically are $\text{C} = 12.007$ and $\text{C} = 12.007$. As the latter figure is quite independent of all previous determinations it has distinct corroborative significance.

Chlorine and Sodium.—In a very elaborate investigation upon the atomic weights of chlorine and sodium, Richards and Wells (published by the Carnegie Institution of Washington, 70 pp., April, 1905; see also *Journ. Am. Chem. Soc.*, 1905, xxvii., 459) have shown that the data furnished by Stas are affected by appreciable errors. Ten syntheses of silver chloride gave, in mean, $\text{Cl} = 35.473$ when $\text{Ag} = 107.93$. From ten measurements of the ratio $\text{Ag} : \text{NaCl}$, and ten of the ratio $\text{AgCl} : \text{NaCl}$, with the foregoing values for silver and chlorine, $\text{Na} = 23.008$. Stas's values are $\text{Cl} = 35.455$ and $\text{Na} = 23.048$.

The results just cited are obviously indirect, for they depend upon the atomic weight of silver. The experiments published by Dixon and Edgar (*CHEMICAL NEWS*, 1905, xci., 263) are therefore of peculiar interest, for they involve the intervention of no intermediate quantities between the atomic weights of chlorine and hydrogen. Hydrochloric acid was directly synthesised from weighed amounts of hydrogen and chlorine, and nine concordant determinations gave, in mean, $\text{Cl} = 35.195$, ± 0.0019 , referred to the hydrogen standard. With $\text{O} = 16$, Cl becomes 35.463, a value nearly midway between that found by Stas and the new figure given by Richards and Wells. Considering the difficulties of the work, the agreement between this and the previous investigations is as close as could be reasonably expected.

Gadolinium.—Urbain (*Comptes Rendus*, 1905, cxl., 583), from ten analyses of the octohydrated sulphate, finds $\text{Gd} = 157.23$; when $\text{H} = 1.007$ and $\text{S} = 32.06$. This value is more than a unit higher than that given in the table, and is probably more trustworthy. (See Eberhard, *Zeit. Anorg. Chem.*, 1905, xlv., 374, on the spectroscopic purity of the rare earth oxides studied by Urbain and others.)

Iodine.—Baxter has continued his research of 1904 upon the atomic weight of iodine, and has published a second memoir upon this subject (*Journ. Am. Chem. Soc.*, 1905, xxvii., 876). First, from eight conversions of silver iodide into bromide by heating in bromine, he found $\text{I} = 126.985$. Two series of measurements, of five experiments in each, of the ratio AgI to AgCl , gave respectively 126.982 and 126.284. Eight determinations of the direct ratio between silver and iodine, weighed separately, gave in mean 126.987. Five determinations of the ratio $\text{I} : \text{AgI}$ gave 126.983, and four of the ratio $\text{Ag} : \text{AgI}$ gave 126.989. The average of all six series is $\text{I} = 126.985$; when $\text{Ag} = 107.93$, $\text{Cl} = 35.473$, and $\text{Br} = 79.955$. The last of these antecedent values was checked by a direct comparison of AgBr with AgCl , and the mean of six experiments gave $\text{Br} = 79.953$. The value previously found by Baxter was 126.975 for

iodine, and the difference is partly due to his use, in the later investigation, of the new figure obtained for chlorine by Richards and Wells. The iodine work, therefore, is confirmatory of the latter.

Nitrogen.—R. W. Gray, in a preliminary notice (*Proc.*, 1905, xxi., 156), gives the results of his experiments upon nitric oxide. Ten determinations of the density of the gas, corrected by D. Berthelot's formula, give a molecular weight of 30.005, whence $\text{N} = 14.005$. Six analyses of nitric oxide, effected by burning finely-divided nickel in it, gave $\text{N} = 14.006$. The investigation was to be continued farther.

Guye, in an interesting lecture before the Chemical Society of Paris (*Bull. Soc. Chem.*, Aug. 5, 1905, with independent pagination; compare Richards, *Proc. Am. Phil. Soc.*, 1904, xliii., 116), has given a complete summary of the researches relative to this atomic weight, which have been conducted by him and his associates at Geneva. He also discusses, somewhat fully, all previous determinations of the constant, and concludes mainly from the physical evidence, that the atomic weight of nitrogen is not far from 14.01, and that the value 14.04, obtained by Stas, is no longer tenable. Going still farther, he reverses the ordinary gravimetric ratios from which the accepted atomic weight of nitrogen was derived, and, applying to them the new value for N , he deduces the atomic weight of silver. The latter is thus reduced from 107.93 to below 107.89, ranging even as low as 107.871. For these low values Guye cites much confirmatory evidence, which is not to be lightly disregarded. To this point we shall recur later.

Potassium.—Atomic weight re-determined by Archibald (*Trans. Roy. Soc. Canada*, Series 2, x., Section iii., p. 47) through analysis of the chloride. Four measurements of the ratio $\text{AgCl} : \text{KCl}$ gave $\text{K} = 39.139$, and four of the ratio $\text{Ag} : \text{KCl}$ gave 39.140; when $\text{Ag} = 107.93$ and $\text{Cl} = 35.455$. If $\text{Cl} = 35.473$, then K becomes 39.122.

Silicon.—W. Becker and J. Meyer (*Zeit. Anorg. Chem.*, 1905, xliii., 251; xlv., 45) have determined the atomic weight of silicon by conversion of the chloride into the oxide. Eight determinations were made, and, in sum, 46.82400 grms. of SiCl_4 gave 16.58236 grms. of SiO_2 . Hence, with $\text{Cl} = 35.45$, $\text{Si} = 28.207$. With the Richards-Wells value for chlorine, 35.473, Si becomes 28.257. Additional determinations by a different method are promised. This paper is preceded by an essay by Meyer on the calculation of atomic weights (*Ibid.*, p. 242), which deserves careful consideration.

Strontium.—From four measurements of the ratio $2\text{Ag} : \text{SrCl}_2$, Richards (*Proc. Am. Acad.*, 1905, xl., 603) finds $\text{Sr} = 87.661$, when $\text{Ag} = 107.93$ and $\text{Cl} = 35.473$. This confirms the earlier value derived by Richards from experiments upon strontium bromide.

Tellurium.—Gallo (*Atti Accad. Lincei*, 1905, [v.], xiv., 23 and 104) have determined electrolytically the ratio between silver and tellurium. From twelve experiments, in mean, $\text{Te} = 127.61$, when $\text{Ag} = 107.93$. Incidentally to this work, as a check upon the method employed, four comparisons between silver and copper were made. In mean, $\text{Cu} = 63.58$.

Thorium.—R. J. Meyer and Gumperz (*Ber.*, 1905, xxxviii., 817) have attempted to separate ordinary thorium into fractions of different atomic weight, and have failed to verify the observations of Baskerville. The fractions obtained by various processes gave atomic weights varying from 232.2 to 232.7, values which are essentially identical with that given in the table.

From the foregoing summary of results, it becomes evident that a far-reaching series of changes will soon be needed in our system of atomic weights. A change in chlorine or nitrogen implies many other changes in the table, and if the accepted value for silver should be modified the alterations would be most sweeping. The atomic weights of silver, chlorine, and bromine enter into the calculation of nearly all other atomic weights, and form, so to speak, the platform upon which the entire structure stands.

The changes, however, which are suggested at present, are not final. Work is in progress in several laboratories which may confirm or modify many of the accepted values, and until that work is finished, at least so far as the fundamental data are concerned, it is wisest for us to suspend judgment and await developments. Were we to reconstruct the table of atomic weights on the basis of the evidence now before us, we should do it imperfectly, and a new revision would be demanded next year or the year after. Confusion would inevitably follow. Fortunately, the matter is not urgent, for the corrections which now seem desirable are not large, and the existing figures are accurate enough for all ordinary purposes. We therefore recommend that the table for 1905 be continued in use without change during 1906, even although some modifications are theoretically desirable. A year hence we shall be in a better position for a critical adjustment of the data, and no harm can follow from the delay. In accordance with the expressed wish of a majority of the larger committee, we also recommend that the table based upon the oxygen standard be made official. So far as this Committee is concerned, the private opinions of its members will be subordinated to the desires of the majority, and the table referred to hydrogen will no longer appear as a part of its report.

For the present, a few suggestions may not be unacceptable, which follow from an examination of the lecture by Guye. Rayleigh, Leduc, Guye, Gray, and others, from their studies of nitrogen and its oxides, have accumulated a mass of strong evidence in favour of a lower value for nitrogen. The data furnished by Stas, on the other hand, point to the higher value which has heretofore been generally adopted. Can we abandon one in the favour of the other, and accept the new figure without reserve?

On behalf of the new figure for nitrogen, we must admit that the determinations are remarkably concordant, and that they rest upon a direct comparison of the element with oxygen. The Stas values, with their confirmations by other chemists, are also very concordant, but they are indirect. They all rest primarily upon the atomic weights of silver, chlorine, and bromine, and these were connected with oxygen through experiments upon chlorates and bromates. Our whole system of atomic weights, with only a few exceptions, is based to-day upon the analyses of several oxyhalogen salts. Their accuracy is assumed, and all anomalies which appear in determinations based upon other lines of research are commonly ascribed to undiscovered errors. The assumption may be sustained, but it is not yet beyond the reach of criticism.

Consider, for example, the well known ratio $\text{Ag} : \text{AgNO}_3 = 100 : 157.149$. If $\text{Ag} = 107.93$, as determined through the analyses of chlorates and bromates, then $\text{N} = 14.037$, or 14.04 as given in our table. If, on the other hand, $\text{N} = 14.009$, as given by Guye, Ag becomes 107.881. The difference between the two values for silver evidently represents a difference in our methods of connecting the element with oxygen, the latter being taken as the standard. For each method strong arguments are possible, and for each value other corroborative testimony can be cited. Neither procedure is wholly free from objections, and the final conclusion, therefore, is one of uncertainty. We cannot safely reject either line of evidence, nor can we accept one as surely more exact than the other. Concordant values for silver can be derived from either method of discussion, as Guye has shown, and through them the entire system of atomic weights is affected.

In this condition of affairs, the position of the Committee can only be one of conservatism. It is better to retain the table we have until at least some of the doubts which now affect it have been eliminated. It seems to be essential that the foundations of the atomic weight table should be both broadened and strengthened, and that new lines of research connecting the fundamental values with oxygen shall be investigated. Some work of this kind is already promised from the laboratory of Harvard University, to be carried out by Richards and his colleagues, but that need

not exclude other activities. It is to be hoped that a number of investigators may take up the consideration of this problem, and that the methods of attack upon it may be multiplied. The careful study of such salts as the sulphates, carbonates, and nitrates might perhaps be profitable. Whether the organic salts of silver could be utilised for good atomic weight determinations is still uncertain.

F. W. CLARKE.
HENRI MOISSAN.
KARL SEUBERT.
T. E. THORPE.

International Atomic Weights: (1906).

		O=16.
Aluminium	Al	27.1
Antimony	Sb	120.2
Argon	A	39.9
Arsenic	As	75.0
Barium	Ba	137.4
Bismuth	Bi	208.5
Boron	B	11
Bromine	Br	79.96
Cadmium	Cd	112.4
Cæsium	Cs	132.9
Calcium	Ca	40.1
Carbon	C	12.00
Cerium	Ce	140.25
Chlorine	Cl	35.45
Chromium	Cr	52.1
Cobalt	Co	59.0
Columbium	Cb	94
Copper	Cu	63.6
Erbium	Er	166
Fluorine	F	19
Gadolinium	Gd	156
Gallium	Ga	70
Germanium	Ge	72.5
Glucinum	Gl	9.1
Gold	Au	197.2
Helium	He	4
Hydrogen	H	1.008
Indium	In	115
Iodine	I	126.97
Iridium	Ir	193.0
Iron	Fe	55.9
Krypton	Kr	81.8
Lanthanum	La	138.9
Lead	Pb	206.9
Lithium	Li	7.03
Magnesium	Mg	24.36
Manganese	Mn	55.0
Mercury	Hg	200.0
Molybdenum	Mo	96.0
Neodymium	Nd	143.6
Neon	Ne	20
Nickel	Ni	58.7
Nitrogen	N	14.04
Osmium	Os	191
Oxygen	O	16.00
Palladium	Pd	106.5
Phosphorus	P	31.0
Platinum	Pt	194.8
Potassium	K	39.15
Praseodymium	Pr	140.5
Radium	Rd	225
Rhodium	Rh	103.0
Rubidium	Rb	85.5
Ruthenium	Ru	101.7
Samarium	Sm	150.3
Scandium	Sc	44.1
Selenium	Se	79.2
Silicon	Si	28.4
Silver	Ag	107.93
Sodium	Na	23.05
Strontium	Sr	87.6

			O=16.
Sulphur	S	32.06	
Tantalum	Ta	183	
Tellurium	Te	127.6	
Terbium	Tb	160	
Thallium	Tl	204.1	
Thorium	Th	232.5	
Thulium	Tm	171	
Tin	Sn	119.0	
Titanium	Ti	48.1	
Tungsten	W	184	
Uranium	U	238.5	
Vanadium	V	51.2	
Xenon	Xe	128	
Ytterbium	Yb	173.0	
Yttrium	Yt	89.0	
Zinc	Zn	65.4	
Zirconium	Zr	90.6	

NOTICES OF BOOKS.

Finite and Infinite. By THOMAS CURRAN RYAN. Philadelphia and London: J. B. Lippincott Company. 1905.

THE author of this work, like so many who make a hobby of science or philosophy without devoting all their energies to it, discusses his subject with great heat and verbosity, although he does not show that he has mastered the essentials of these branches of knowledge. This is specially noticeable in the second part of the book, in which an endeavour is made to prove that the universe is finite—a good many facts and arguments being brought forward to demonstrate also that the sky is matter of some sort beyond the stars. The author expresses considerable surprise that the editor of "Popular Astronomy," to whom he had entrusted an article containing an exposition of his theories, did not make use of it; but it may be suggested that possibly an explanation is to be found in the fact that these views, when original, are hardly satisfactorily established in his work. The first part of the book discusses Idealistic Pantheism, with special reference to Fiske's writings, which are criticised at some length, the object being to warn the Christian world of the danger threatened to it by Idealism.

Investigations of Infra-red Spectra. By WILLIAM W. COBLENTZ. Washington, D.C.: The Carnegie Institution of Washington. 1905.

THIS work gives a most complete account of the systematic investigation of infra-red spectra, begun by the author when a student at Cornell University and continued at the Carnegie Institution of Washington. Part I. of the book deals with the Infra-red Absorption Spectra, and describes researches undertaken for the purpose of determining the effect of molecular weight upon absorption spectra. After an explanation of the method and apparatus employed, the results obtained with at least 130 compounds of carbon and hydrogen, the spectra of which were explored in the majority of cases to $14\ \mu$ or $15\ \mu$, are described, and a general discussion of the whole work draws attention to various important conclusions which may be drawn from it. This part of the book also contains many tables of line spectra, besides transmission curves. Part II. gives a record of a research on the Infra-red Emission Spectra, the object of the work being the study of the region lying beyond $2\ \mu$. This section is comparatively short, and consists chiefly of an account of experimental observations, although some theoretical points are briefly discussed. The whole book is most interesting, and spectroscopists will find that it well deserves careful reading.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

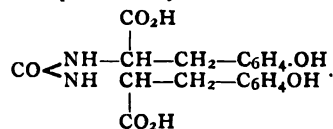
Comptes Rendus Hebdomadaires des Séances de l'Académie des Sciences. Vol. cxlii., No. 1, January 2, 1906.

Estimation of Carbonic Oxide in the Air by means of Iodic Anhydride.—Armand Gautier.—The author adds as a note to Jaubert's paper in the last number that as long ago as 1898 he noticed that the method of estimating the carbonic oxide in the air by means of iodic anhydride, contains an error due to the fact of the reaction of the latter substance on acetylene. He also gave a means of correcting the error. He states, however, that acetylene is not present in appreciable quantities in ordinary town air.

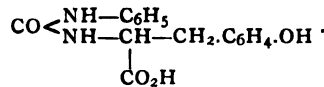
Determination of Rare Gases in Natural Gaseous Mixtures.—Charles Moureu.—The author describes with drawing the apparatus he is using in a series of researches on the determination of rare gases in natural gaseous mixtures.

Heat of Fusion of Ice.—A. Leduc.—The author re-determines the heat of fusion of ice, and after careful experiments and calculation finds the specific mass of ice at 0° to be 0.9176 instead of 0.91674 as determined by Bunsen. From this the heat of fusion of ice is found to be 79.2 calories at 15° .

Synthetic Union of the Amide Acids Derived from the Albumines.—L. Hugounenq and A. Morel.—The authors' researches on the synthetic union of the amide acids derived from the albumines were made on tyrosine, which is with leucine one of the most important products of the decomposition of albumenoid bodies. The researches lead to the following results:—1. Below 100° carbon oxychloride has no action on tyrosine. At about 150° in a sealed tube the reaction is accompanied by the formation of tarry products. 2. In the cold, COCl_2 acts on the sodium salt of tyrosine dissolved in water, giving a symmetric urea product of tyrosine,—



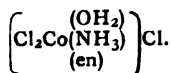
The carbimides react on tyrosine dissolved in alkaline water, giving rise to mixed urea products of tyrosine and amines, and so leading to the preparation of mixed urates of the two different amide acids. As an example, the author prepares the mixed urea product of tyrosine and aniline,—



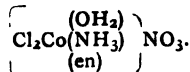
Berichte der Deutschen Chemischen Gesellschaft.
Vol. xxxviii., No. 17, 1905.

Mixed Triamine Cobalt Salts containing Ethylene Diamine and Ammonia.—A. Werner and A. Grün.—The investigation of some members of the triamine cobalt series containing ethylene diamine has shown that the presence of ethylene diamine alters the chemical and physical properties of the triamine salts more than is the case with tetramine salts, and that, in contrast to the behaviour of the tetramine salts, the stability of the cobaltic radical of the triamine salts is not raised but

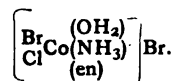
lowered by the presence of ethylene diamine. Trinitro ethylene diamine triamine cobalt, formed by the action of ethylene diamine on the tetranitro diamine cobalt acid salt, is converted by boiling hydrochloric acid into a black salt, which, on re-crystallisation from water containing hydrochloric acid, gives greenish black needles of composition:—



Only one chlorine atom of this salt is replaced in the usual way; thus when nitric acid acts upon it the following nitrate is formed:—



In aqueous solution one intra radical chlorine atom, in consequence of hydration, suffers a very rapid electrolytic dissociation, so that the green solution turns blue. This blue solution yields olive-green needles when hydrobromic acid is added, and the compound thus formed has the formula—

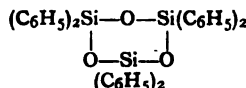


These transformations show that the presence of ethylene diamine in the triamine cobalt radical increases the mobility of the acid residues combined with the cobalt, so that the internal stability of the complex cobalt radicals in the form of salts is much less than that of the ordinary triamine salts.

Perchromic Acid Anhydride Triamine.—E. H. Riesenfeld.—Hofmann and Hiendlmaier (*Berichte*, 1905, xxxviii., 3059; *CHEMICAL NEWS*, 1905, xcii., 201) think that when chromate is oxidised by hydrogen peroxide the composition of the product varies according to the amount of ammonia present in the solution. The author's experiments, however, show that whether the ammonium chromate solution contains only 2.5 per cent free ammonia, or is saturated with ammonia, an ammonium perchromate of the formula $(\text{NH}_4)_3\text{CrO}_8$ is obtained, provided that the solution contains a sufficient quantity of hydrogen peroxide (3 to 5 per cent), and that it is not warmed above 0° until all the salt has completely crystallised out. The form of the crystals, however, is dependent upon the amount of ammonia present. If the solution contains only small amounts of ammonia, octohedric pyramids are obtained, and when large amounts of ammonia are present the crystals which separate out are identical with those which Hofmann and Hiendlmaier obtained as impurities in the preparation of perchromic acid anhydride triamine. If the solution contains an insufficient amount of hydrogen peroxide, or is warmed above 0° , either a mixture of $(\text{NH}_4)_3\text{CrO}_8$ and $\text{CrO}_4 \cdot 3\text{NH}_3$ is obtained, or else the latter only. This is due to the fact that perchromic acid anhydride triamine is much more stable than ammonium perchromate. Perchromic acid anhydride triamine is known in the form of needles and plates, and Hofmann and Hiendlmaier thought that these were two isomeric substances. Crystallographic examination, however, shows that they are not isomeric forms, but are probably identical. When the triamine is decomposed by means of acids chromic peroxide is not formed, but a mixture of chromium oxide and hydrogen peroxide.

Diphenyl Silicon Oxide and Benzyl Silicon Compounds.—W. Dilthey.—By the action of phenyl magnesium bromide on silicon tetrachloride in ethereal solution mono-, di-, or tri-phenyl silicon compounds are formed, according to the amount of magnesium compound used. It has not yet been found possible to replace the fourth atom of chlorine by the phenyl group. Diphenyl silicon hydroxide, $(\text{C}_6\text{H}_5)_2\text{Si}(\text{OH})_2$, on being fused yields diphenyl silicon oxide, $(\text{C}_6\text{H}_5)_2\text{SiO}$. The latter forms a gelatinous

mass, but may be obtained in crystalline form (melting at 188°) by adding a drop of acetic acid anhydride to the gelatine, when the whole mass instantly crystallises. The determination of the molecular weight of the crystals cryoscopically in alcohol showed that the formula $(\text{C}_6\text{H}_5)_2\text{SiO}$ must be trebled, while approximately the same result was obtained from the gelatine in benzene. As diphenyl silicon hydroxide, $(\text{C}_6\text{H}_5)_2\text{Si}(\text{OH})_2$, is monomolecular, evidently at first the gelatine is monomolecular, but is immediately transformed to the trimolecular form, which then crystallises. This behaviour recalls the frequently occurring polymerisation phenomena which have been observed with carbon non-metallic compounds. The constitutional formula is—

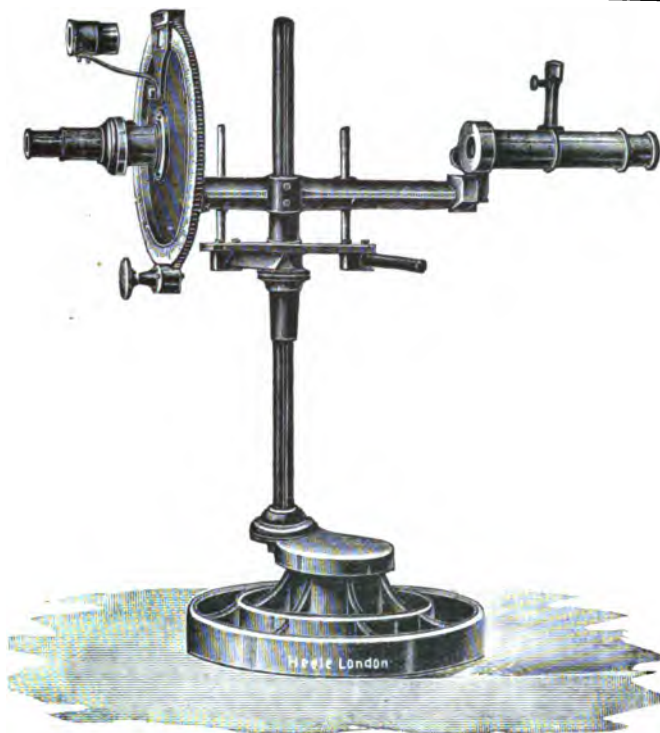


Diphenyl silicon oxide is the first substance (as far as the author knows) in which the power of polymerisation has been experimentally demonstrated, although the constitutional formulæ which have been adopted for the silicates have illustrated the same great tendency to form condensed molecules.

Electrolytic Reduction of Carbonic Acid.—R. Ehrenfeld.—It has been shown that when cold saturated potassium sulphate solutions are electrolysed with a low current density, a current of carbon dioxide being steadily led through them, formic acid appears in the cathode liquid, and it is the HCO_3 ion which undergoes the reduction. The author has electrolysed ammonium carbonate solutions, and examined the reduction products resulting at the amalgamated zinc cathode. He finds that when the cathode liquid is distilled in a current of steam, formic acid can be detected in the distillate. No reduction occurs when the cathode is iron, platinum, copper, lead, or nickel. If either the concentration of the ammonium carbonate solution or the current density sinks below a certain minimum no reduction occurs. It is the NH_4CO_3 ion which is reduced at the cathode, for formic acid is formed when solutions of ammonium carbonate containing large amounts of ammonia are electrolysed. A difficulty arises, however, when it is observed that no formic acid is formed when cold saturated sodium carbonate solutions are electrolysed, but it may be that other factors condition the electrolytic formation of formic acid from salts of CO_2 although the HCO_3 ion is at the basis of the reduction.

MEETINGS FOR THE WEEK.

- MONDAY, 5th**—Society of Arts, 8. (Cantor Lectures). "Modern Warships," by Sir William White, F.R.S., &c.
— Royal Institution, 5. General Monthly Meeting.
— Society of Chemical Industry, 8. "Loss of Nitre in the Chamber Process" (Part II.), by J. K. H. Inglis.
- TUESDAY, 6th**—Royal Institution, 5. "Food and Nutrition," by Prof. William Stirling, M.D., &c.
— Society of Arts, 4.30. "Imperial Immigration," by Octavius Charles Beale.
- WEDNESDAY, 7th**—Society of Arts, 8. "Progress in Electric Lighting," by Leon Gaster.
— Society of Public Analysts, 8. (Annual Meeting). President's Address. "Note on Dutch Cheese," by C. H. Cribb. "Assay of Mercury (ores)," by G. T. Holloway. "Purification of Zinc and Hydrochloric Acid," by L. T. Thorne and E. H. Jeffers. "The Facing of Rice," by C. H. Cribb and P. A. E. Richards.
- THURSDAY, 8th**—Royal Institution, 5. "The Significance of the Future in the Theory of Evolution," by Benjamin Kidd.
- FRIDAY, 9th**—Royal Institution, 9. "Eclipse Problems and Observations," by Hugh Frank Newall, F.R.S., &c.
— Physical 8. (Annual General Meeting). Address by Prof. Perry, President-Elect.
- SATURDAY, 10th**—Royal Institution, 3. "Advances in Microscopy," by John W. Gordon.



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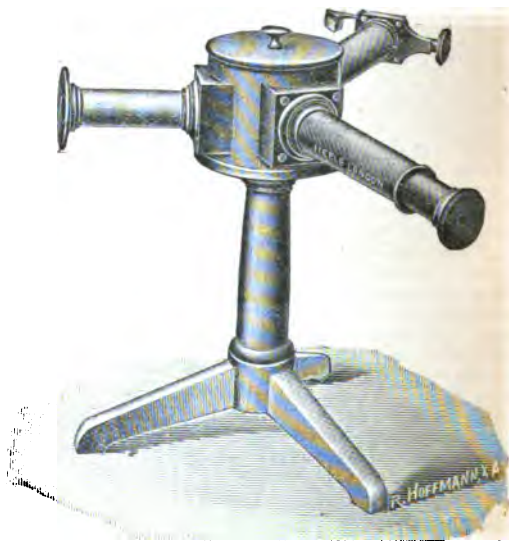
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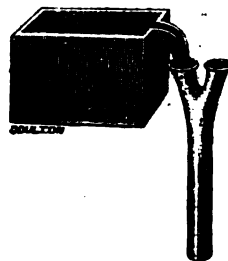
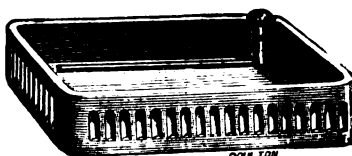
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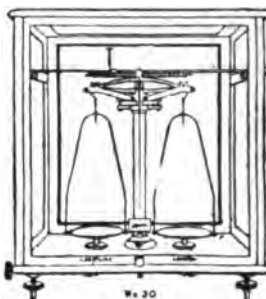
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THE INTERACTION OF HYDROCHLORIC ACID AND POTASSIUM PERMANGANATE IN THE PRESENCE OF FERRIC CHLORIDE.*

By JAMES BROWN.

LÖWENTHAL (*Zeit. Anal. Chem.*, i., 329) and Lenssen were the first to show that the titration of ferrous salts by potassium permanganate in the presence of hydrochloric acid, as proposed by Margueritte (*Ann. Chim.*, [3], xviii., 244), does not admit of quantitative accuracy because of the evolution of chlorine by the interaction of hydrochloric acid and potassium permanganate, and to propose as a remedy for this source of error the titration of successive equal portions of the ferrous salt to be determined until the readings become constant.

This tendency toward evolution of chlorine in titrations of a ferrous salt by potassium permanganate in the presence of hydrochloric acid, as compared with the alleged absence of such tendency in similar titrations of oxalic acid, was explained by Zimmermann on the supposition that the oxidation of the iron proceeds so rapidly as to form oxides of iron higher than the sesquioxide, which then react to oxidise more iron and liberate chlorine from hydrochloric acid (*Ann. Chem.*, ccxiii., 311). Quite recently Wagner explains this phenomenon by the assumed formation of chlor-ferrous acid (analogous to chlor-platinic and chlor-auric acids), which is more easily oxidised by the permanganate than is hydrochloric acid under similar conditions ("Maassanalytische Studien, Habilitationsschrift," Leipzig, 1898). Recent work has shown that there is a slight though real waste of permanganate in titrations of oxalic acid under the conditions named, and that this loss is proportional to the amount of hydrochloric acid present (Gooch and Peters, *Am. Journ. Sci.*, [4], vol. vii., 463). It still appears, however, that this loss is greater in titrations of ferrous salts than in those of oxalic acid under the conditions named.

Wagner's work in relation to the phenomenon mentioned above has been reviewed very carefully, and it has been found that, although as shown by him more permanganate is required to bring about final coloration against equal quantities of oxalic acid in experiments in which equal quantities of potassium permanganate are digested with a constant quantity of normal hydrochloric acid and a measured volume of tenth normal ferric chloride, than when an equivalent quantity of tenth-normal hydrochloric acid is substituted for the tenth-normal ferric chloride, the differences vary within wide limits and disappear entirely if the chlorine formed by the interaction of the potassium permanganate and hydrochloric acid is removed during the digestion. When also the chlorine is thus removed the same quantity of permanganate is required to bring about final coloration, whether ferric chloride is present or not. It is found also that the permanganate is entirely destroyed within the limits proposed by Wagner, and that after an hour's digestion, and in fact long before, the permanganate colour has entirely disappeared and only the hydrated oxides of manganese—formed according to the Guyard reaction—are visible in the digestion liquid.

Wagner describes no special form of apparatus in his work, and gives no details as to size of flask used to contain the digestion liquids, form of bath, &c., pointing out the fact simply that he used a return-condenser 60 c.m. in

length. It was found convenient in the experiments about to be described to use a 250 c.c. flask to contain the solutions during digestion, and to heat the solutions in an Ostwald thermostat.

The experiments of Table I. were conducted, as outlined by Wagner, in the following manner:—To a 250 c.c. flask were added 100 c.c. of normal hydrochloric acid (that is, a solution containing 36.4575 grms. of the acid to the litre), and in addition either 9.91 c.c. of tenth-normal hydrochloric acid (prepared by diluting 100 c.c. of the normal solution to one litre) or 9.91 c.c. of tenth normal ferric chloride. Of approximately twentieth normal potassium permanganate, carefully standardised against ammonium oxalate, 9.91 c.c. were then added, and the flask, fitted in a ground joint to a return-condenser 60 c.m. in length and with a bore approximately 3 c.m. in diameter, was heated for one hour in the Ostwald thermostat at a temperature of 50° C. Of tenth normal oxalic acid, 9.91 c.c. were then added to the digestion liquid and a measured volume of the same permanganate solution as was added before digestion was run in to colour. The difference between the total permanganate used (that is, the permanganate added before digestion plus that added to bring about final coloration against the oxalic acid) and the permanganate equivalent of the oxalic acid added, gives, according to Wagner, the permanganate reduced during the digestion. The results of these experiments are recorded in Table I.

Here it may be seen that although in general more permanganate is required to bring about final coloration in those experiments in which ferric chloride was used than in its absence, the results show at best wide variation among themselves, and the amounts of permanganate apparently destroyed during the digestion are at all events considerably greater than in the experiments conducted by Wagner under similar conditions. In the experiments recorded in Table I., in the average 4.73 c.c. of permanganate were apparently destroyed where ferric chloride was not used, and 5.37 c.c. in the presence of ferric chloride; while in Wagner's experiments 0.96 c.c. of permanganate was apparently reduced without use of ferric chloride and 1.41 c.c. in its presence.

Since, as has been noted above, the permanganate colour entirely disappeared in the experiments of Table I. long before the termination of the hour's digestion, while only small amounts of hydrated oxides of manganese varying in colour from brown to black were visible in the digestion liquid, it seemed probable that more permanganate was really reduced during the digestion than is indicated in these experiments. Moreover, a strong odour of chlorine was noticeable in these experiments, and it seemed probable that some of the chlorine, formed by the interaction of the potassium permanganate and hydrochloric acid during the digestion, remained to take part in the oxidation of the oxalic acid introduced, and that, therefore, on running in permanganate solution to colour, less of the latter was required than corresponded to the oxalic acid left unoxidised by the residual oxides of manganese. It was therefore decided to remove if possible this chlorine, and to this end a vigorous current of carbon dioxide or air was passed through the digestion liquid while heating. In this way the chlorine was readily removed and starch and potassium iodide paper held in the current of carbon dioxide or air gave no test for chlorine.

The experiments of Table II., in which no ferric chloride was used, were conducted precisely as were those of Table I., except that a vigorous current of carbon dioxide generated in a Kipp generator by action of hydrochloric acid on marble, and washed and dried by passing first through a bottle filled with water and then through a calcium chloride tube, was passed through the liquid during the process of digestion. Because also of the greater ease of measuring out accurately 9.90 c.c. rather than the 9.91 c.c. used by Wagner and in the experiments of Table I., the former volume of reagents was substituted for the latter in the experiments to follow. It will readily be

* Contributions from the Kent Chemical Laboratory of Yale University. From the *American Journal of Science*, xix., January, 1905.

TABLE I.
(Temperature, 50° C.; Time of Digestion, Sixty Minutes).
9.91 c.c. $\text{H}_2\text{C}_2\text{O}_4 = 20.25$ c.c. KMnO_4 .

N/1 HCl. C.c.	N/10 HCl. C.c.	N/10 FeCl_3 . C.c.	KMnO_4 before digestion. C.c.	N/10 $\text{H}_2\text{C}_2\text{O}_4$. C.c.	KMnO_4 to colour. C.c.	KMnO_4 apparently reduced during digestion. C.c.
100	9.91	—	9.91	9.91	15.89	5.55
100	9.91	—	9.91	9.91	15.11	4.77
100	9.91	—	9.91	9.91	15.15	4.81
100	9.91	—	9.91	9.91	15.07	4.73
100	9.91	—	9.91	9.91	15.13	4.79
100	9.91	—	9.91	9.91	15.07	4.73
100	9.91	—	9.91	9.91	15.08	4.74
100	9.91	—	9.91	9.91	15.02	4.68
100	9.91	—	9.91	9.91	14.85	4.51
100	9.91	—	9.91	9.91	14.40	4.06
100	9.91	—	9.91	9.91	15.05	4.71
100	—	9.91	9.91	9.91	15.60	5.26
100	—	9.91	9.91	9.91	15.35	5.01
100	—	9.91	9.91	9.91	15.32	4.98
100	—	9.91	9.91	9.91	15.88	5.54
100	—	9.91	9.91	9.91	15.42	5.08
100	—	9.91	9.91	9.91	15.45	5.11
100	—	9.91	9.91	9.91	15.95	5.61
100	—	9.91	9.91	9.91	15.41	5.07
100	—	9.91	9.91	9.91	15.95	5.61
100	—	9.91	9.91	9.91	15.65	6.31
100	—	9.91	9.91	9.91	15.75	5.41
100	—	9.91	9.91	9.91	15.79	5.45

TABLE II.
(Temperature, 50° C.; Time of Digestion, Sixty Minutes).

	N/1 HCl. C.c.	N/10 HCl. C.c.	KMnO_4 before digestion. C.c.	$\text{H}_2\text{C}_2\text{O}_4$. C.c.	KMnO_4 to colour. C.c.	KMnO_4 apparently reduced during digestion. C.c.
9.90 c.c. approximately N/10 $\text{H}_2\text{C}_2\text{O}_4 = 23.52$ c.c. KMnO_4 .						
I.	100	9.90	9.90	9.90	22.19	8.57
II.	100	9.90	9.90	9.90	22.10	8.48
III.	100	9.90	9.90	9.90	22.15	8.53
IV.	100	9.90	9.90	9.90	22.11	8.49
9.90 c.c. approximately N/10 $\text{H}_2\text{C}_2\text{O}_4 = 23.65$ c.c. KMnO_4 .						
V.	100	9.90	9.90	9.90	22.15	8.40
VI.	100	9.90	9.90	9.90	21.79	8.04
VII.	100	9.90	9.90	9.90	22.24	8.49
VIII.	100	9.90	9.90	9.90	22.21	8.46
IX.	100	9.90	9.90	9.90	22.24	8.49
X.	100	9.90	9.90	9.90	22.23	8.48
XI.	100	9.90	9.90	9.90	22.25	8.50
XII.	100	9.00	9.90	9.90	22.15	8.40
XIII.	100	9.90	19.80	9.90	19.77	15.92
XIV.	100	9.90	19.80	9.90	19.83	15.98
XV.	100	9.90	50.0	25.00	44.53	34.81
XVI.	100	9.90	50.0	25.00	44.47	34.75
XVII.	100	9.90	50.0	25.00	44.44	34.72
XVIII.	100	9.90	50.0	25.00	44.58	34.86

seen that in the case of tenth normal hydrochloric acid 0.01 c.c. is negligible as compared with the large amount of hydrochloric acid used in the experiments.

The results of these experiments are recorded in Table II.

From these results the conclusion may be drawn that the low indications of the amount of permanganate apparently reduced during digestion in the experiments recorded in Table I., at least so far as concerns those experiments in which no ferric chloride was used, were in all probability due to the oxidising action of the unexpelled chlorine on the oxalic acid, and that the large variations in results were due to the greater or less retention of the chlorine. In Experiments XIII. to XVIII. it is seen further that amounts

of permanganate very much greater than those used in Wagner's experiments and in the experiments of Table I. can be reduced by the same amount of hydrochloric acid, and under the same conditions of temperature and time as in those other experiments; for, in these last experiments, also, the permanganate colour entirely disappeared during the digestion.

In order to ascertain if a current of air is equally as effective in removing the chlorine as is carbon dioxide, and also because of the greater availability of the former, the experiments recorded in Table III. were conducted in a manner identical with those of Table II., except that a current of air dried and purified was substituted for the carbon dioxide. When also the success of the air current

TABLE III.

(Temperature, 50° C.).

9.90 c.c. approximately N/10 $\text{H}_2\text{C}_2\text{O}_4$ = 20.09 c.c. KMnO_4 .

	N/1 HCl.	N/10 HCl.	N/10 FeCl ₃ .	KMnO ₄ before digestion.	Time of digestion.	Residual KMnO ₄ colour after digestion.	Cl test after digestion.	H ₂ C ₂ O ₄ .	KMnO ₄ to colour.	KMnO ₄ apparently reduced during digestion.
	C.c.	C.c.	C.c.	C.c.	Mins.			C.c.	C.c.	C.c.
I.	100	9.90	—	9.90	60	None	None	9.90	18.88	8.69
II.	100	9.90	—	9.90	60	"	"	9.90	18.87	8.68
III.	100	9.90	—	9.90	60	"	"	9.90	18.80	8.61
IV.	100	9.90	—	9.90	60	"	"	9.90	18.81	8.62
V.	100	9.90	—	9.90	60	"	"	9.90	18.80	8.61
VI.	100	9.90	—	9.90	60	"	"	9.90	18.82	8.63
VII.	100	9.90	—	9.90	30	"	"	9.90	18.77	8.58
VIII.	100	9.90	—	9.90	30	"	Doubtful	9.00	18.70	8.51
IX.	100	9.90	—	9.90	15	"	Very faint	9.90	18.70	8.51
X.	100	9.90	—	9.90	15	"	"	9.90	18.68	8.49
XI.	100	—	9.90	9.90	60	"	None	9.90	18.87	8.68
XII.	100	—	9.90	9.90	60	"	"	9.90	18.85	8.66
XIII.	100	—	9.90	9.90	60	"	"	9.90	18.81	8.62
XIV.	100	—	9.90	9.90	60	"	"	9.90	18.81	8.62
XV.	100	—	9.90	9.90	60	"	"	9.90	18.87	8.68
XVI.	100	—	9.90	9.90	60	"	"	9.99	18.85	8.66
XVII.	100	—	9.90	9.90	30	"	"	9.90	18.80	8.61
XVIII.	100	—	9.90	9.90	30	"	Doubtful	9.90	18.72	8.53
XIX.	100	—	9.90	9.90	15	"	Very faint	9.90	18.65	8.46
XX.	100	—	9.90	9.90	15	"	"	9.90	18.67	8.48

TABLE IV.

(Temperature, 50° C.).

	N/1 HCl.	N/10 HCl.	KMnO ₄ before digestion.	H ₂ O.	Volume during digestion.	Time of diges- tion.	Residual KMnO ₄ colour after digestion.	Cl test after digestion.	H ₂ C ₂ O ₄ .	KMnO ₄ to colour.	KMnO ₄ apparently reduced during digestion.
	C.c.	C.c.	C.c.	C.c.	C.c.	Mins.			C.c.	C.c.	C.c.
40 c.c. N/10 $(\text{H}_4\text{N})_2\text{C}_2\text{O}_4$ = 37.64 c.c. KMnO_4 ; 100 c.c. $\text{H}_2\text{C}_2\text{O}_4$ = 101.40 c.c. KMnO_4 .											
I.	100	9.90	100	—	210	60	None	Faint	100	59.52	58.12
II.	100	9.90	100	—	210	60	"	"	100	58.44	57.04
40 c.c. N/10 $(\text{H}_4\text{N})_2\text{C}_2\text{O}_4$ = 18.35 c.c. KMnO_4 ; 50 c.c. N/5 $\text{H}_2\text{C}_2\text{O}_4$ = 49.26 c.c. KMnO_4 .											
III.	100	9.90	50	—	160	60	None	None	50	30.72	31.46
IV.	100	9.90	50	—	160	60	"	"	50	30.75	31.49
V.	100	9.90	50	—	160	60	"	"	50	30.76	31.50
VI.	100	9.90	50	—	160	60	"	"	50	30.78	31.52
VII.	100	9.90	50	—	160	60	"	"	50	30.88	31.62
VIII.	100	9.90	50	50	210	60	Faint	Faint	50	27.11	27.85
IX.	100	9.90	50	50	210	60	"	"	50	27.92	28.66
X.	100	9.90	50	50	210	60	"	"	50	28.52	29.26
XI.	100	9.90	50	50	210	60	"	"	50	28.54	29.28
XII.	100	9.90	50	50	210	60	"	"	50	29.14	29.88
XIII.	100	9.90	50	50	210	60	"	"	50	28.98	29.72
XIV.	100	9.90	50	50	210	60	"	"	50	28.56	29.30
XV.	100	9.90	50	50	210	60	"	"	50	29.96	30.70
XVI.	100	9.90	50	50	210	85	None	Very faint	50	29.96	30.70
XVII.	100	9.90	50	50	210	60	Faint	Faint	50	30.22	30.96
XVIII.	100	9.90	75	—	185	60	None	Marked	50	20.72	46.46
XIX.	100	9.90	50	50	210	120	"	None	50	30.31	31.05
XX.	100	9.90	100	—	210	60	Marked	Marked	60	19.58	60.47
XXI.	100	9.90	75	50	235	220	None	Faint	50	21.84	47.58
XXII.	100	9.90	100	50	260	180	Marked	Marked	60	10.18	51.07
2N/1 HCl.											
XXIII.	50	9.90	50	—	110	60	None	Faint	50	30.23	30.97
XXIV.	50	9.90	75	—	135	60	"	Marked	—	—	—
XXV.	50	9.90	75	—	135	60	"	"	50	30.69	56.43

was apparent, ferric chloride was again used and the effect noted.

Results are outlined in Table III.

Here again may be noted the concordance of results when the chlorine is all removed before the addition of oxalic acid, as well as the fact that under these conditions substantially the same amount of permanganate is required

to bring about the end reaction, whether ferric chloride is present or not; and that consequently as much permanganate is reduced during the digestion in the latter case as in the former. Also by a comparison of Experiments VIII.—X. and XVIII.—XX., in which a slight trace of chlorine remained, with Experiments I.—VI. and XI.—XVI., in which the chlorine was entirely removed,

we again see the oxidising effect of the residual chlorine on the oxalic acid; for even in the former sets of experiments, in which the digestion was carried on only fifteen or thirty minutes, the permanganate colour had entirely disappeared at the end of the digestion. The variations in the amount of permanganate apparently reduced during the digestion in the experiments recorded in Table I. are, therefore, doubtless due to the interfering action of the residual chlorine held in solution. The " KMnO_4 apparently reduced during digestion" in the experiments of Table II., and in those of Table III. in which the chlorine was entirely removed during the digestion, represents the amounts of permanganate entirely reduced to manganous chloride, while the differences between these amounts and the " KMnO_4 before digestion" represent the residual oxides of manganese. Similar differences in the experiments of Table I., and in those of Table III. in which the chlorine was only partially removed, represent the residual oxides of manganese and the chlorine retained in solution.

The amount of chlorine held in solution when no means are employed to remove it, depends largely on the form and size of the flask used to contain the solutions during digestion, also on the dimensions of the return-condenser, and will vary according to the greater or less amount of shaking to which the flask is subjected during the entire course of the experiment. It is, therefore, evident that Wagner's experiments are in no way indicative of the relative amounts of potassium permanganate reduced in the presence or absence of ferric chloride, other conditions being constant, but are an indication simply of the greater or less retention of chlorine in solution in the form of apparatus used by him; for it has been shown that in all experiments conducted within the limits proposed by Wagner the permanganate is entirely destroyed, and that any variations in the amount of permanganate apparently destroyed during digestion disappear when the chlorine is entirely removed from the sphere of action. The possibility of any interfering action of ferric chloride in titrations of oxalic acid by potassium permanganate is excluded by the results of the experiments of Table III., in which we find no variations in results whether ferric chloride is present or not. The cause of the apparently greater destruction of potassium permanganate in those experiments of Table I. in which ferric chloride was used than in those in which ferric chloride was not used, is now under investigation.

Since in all experiments thus far conducted the permanganate colour has been entirely destroyed, the experiments of Table IV. were made to ascertain if possible how much permanganate can be destroyed by the amount of hydrochloric acid used in the experiments of Tables I., II., and III. under the same conditions of time and temperature, and also during greater periods of time. It will readily be seen from the evident oxidation of oxalic acid by chlorine in previous experiments that an exact measure of the maximum amount of permanganate reduction during a given period of time can be obtained only when all the chlorine is removed and at the same time the permanganate colour just disappears—a condition difficult to attain. The results recorded in Table IV. should therefore be regarded as approximate only.

Thus it may be seen that the same amount of hydrochloric acid as was used in the experiments of Tables I., II., and III. is capable of breaking down approximately thirty times as much permanganate as was used in those experiments and in the experiments of Wagner, conditions of time and temperature being the same. Changes of volume are of course involved in the use of varying amounts of permanganate, but an increase in volume would in all probability be attended by a decrease in the relative amount of permanganate reduced by a constant quantity of hydrochloric acid. In any case, the results show a more extensive reduction than is indicated in Wagner's experiments and in those of Tables I., II., and III.

The conclusion must be drawn, then, that Wagner's experiments in no way show the catalytic effect of ferric chloride in the interaction between hydrochloric acid and potassium permanganate, nor do they furnish evidence in support of the assumed formation of chlor-ferrous acid. They afford simply an indication of the greater or less retention of chlorine in solution, and the greater or less oxidising action of this chlorine on the oxalic acid in the presence or absence of ferric chloride.

The author is indebted to Prof. F. A. Gooch for much advice and assistance in the preparation of this paper.

A CONVENIENT APPARATUS FOR DETERMINING VOLATILE SUBSTANCES BY LOSS OF WEIGHT.*

By J. LEHN KREIDER.

Of the various forms of apparatus designed for the determination of volatile products of reactions, some are cumbersome, and many require for their construction skill in glass-blowing. The apparatus here described is light

and easily made from three test-tubes, modified and fitted as shown in the figure. The test-tube, A, is changed in no way from its original form; B is perforated, in the bottom, with a hole about 1 c.m. in diameter, and fits tightly within A; and C, so selected that it fits loosely within B, is drawn out to a small capillary tube.

When the apparatus is to be used, the capillary of C is pushed through the hole of B, packed loosely with cotton; B is filled to the depth of from 6 c.m. to 8 c.m. (about two-thirds of its contents), with granular calcium chloride; and B and C are adjusted as shown.

To the test-tube, C, is fitted a one-holed stopper, through which passes a short glass tube which is to be closed by a rubber cap and plug. Upon removing the plug, and applying suction to the short tube, the reagent employed to liberate the volatile product to be determined is drawn up through this capillary until C is sufficiently filled. Upon replacing the plug the reagent remains within C, held by atmospheric pressure.

The tubes A and B may be so selected that very little of the product evolved can escape between them, but in case they fit very loosely, a ring of paraffin melted into the mouth of A, about B, by means of a hot iron or wire, seals the joint securely. A very convenient way to attach the paraffin is to melt it between A and another tube, which fits A, as does B, and may be removed by a turning motion, leaving the ring into which B will fit, and which then requires very little heating to make a tight joint. If care be used in taking apart A and B, at the close of an experiment, such a ring of paraffin remains in place, and may be used many times without replacement, being re-melted by a touch of the hot wire before every new experiment.

In making a determination, the substance under examination is weighed and placed in the bottom of A. The reagent to be employed, 10 c.m.³ to 15 c.m.³, is drawn into C, and held there in the manner described. The test-tube A is slipped over B, and this joint is sealed with paraffin, as has been shown. The apparatus is wiped, placed on the balance, and weighed.

Upon removing the cap from the small tube in C, the reagent runs from C into A. The volatile product is formed, is forced upward through the drying column of calcium chloride, and escapes through the annular space between

* Contribution from the Kent Chemical Laboratory of Yale University. From the *American Journal of Science*, 1905, xix., p. 188.



a and c. When the action ceases, a current of dry air is forced through c, to drive all the volatile product from the apparatus; the cap is then replaced, and the whole placed on the balance to be weighed. The loss of weight represents the volatile product.

The following results show the accuracy which may be expected when carbonates are treated in the apparatus with dilute hydrochloric acid:—

TABLE I.

Taken. Grm.	Found. Grm.	Error. Grm.
<i>Calcium Carbonate.</i>		
0.2000	0.0879	-0.0001
0.2000	0.0878	-0.0002
0.2000	0.0879	-0.0001
0.2000	0.0879	-0.0001
0.5000	0.2197	-0.0003
0.5000	0.2196	-0.0004
0.5000	0.2194	-0.0006
0.5000	0.2198	-0.0002
0.5000	0.2197	-0.0003
0.5000	0.2197	-0.0003
<i>Barium Carbonate.</i>		
0.5000	0.1134	-0.0011
0.5000	0.1137	-0.0005
0.5000	0.1137	-0.0005
0.5000	0.1136	-0.0006
<i>Strontium Carbonate.</i>		
0.5000	0.1485	-0.0004
0.5000	0.1486	-0.0003
0.5000	0.1485	-0.0004

TABLE II.

Taken. Grm.	Found. Grm.	Error. Grm.
<i>Magnesium Metal.</i>		
0.1000	0.0087	+0.0003
0.1000	0.0085	+0.0001
0.1000	0.0084	0.0000
0.1000	0.0084	0.0000
0.1000	0.0083	-0.0001
<i>Zinc Metal.</i>		
0.2000	0.0061	0.0000
0.2000	0.0062	+0.0001
0.2000	0.0062	+0.0001
0.2000	0.0060	-0.0001
0.2000	0.0061	0.0000

TABLE III.

Taken. Grm.	Found. Grm.	Error. Grm.
<i>Urea.</i>		
0.1000	0.0469	+0.0003
0.1000	0.0467	+0.0001
0.1000	0.0467	+0.0001
0.1000	0.0468	+0.0002
0.1000	0.0467	+0.0001
<i>Ammonium Oxalate.</i>		
0.1000	0.0204	+0.0007
0.1000	0.0197	0.0000
0.1000	0.0198	+0.0001
0.1000	0.0198	+0.0001
0.1000	0.0196	-0.0001
<i>Ammonium Chloride.</i>		
0.1000	0.0264	+0.0002
0.1000	0.0265	+0.0003
0.1000	0.0261	-0.0001
0.1000	0.0263	+0.0001
0.1000	0.0261	-0.0001

In Table II. are included the results of some experiments made to determine the weights of hydrogen liberated by the action of magnesium and zinc, upon dilute hydrochloric acid.

Determinations of the nitrogen liberated from urea, ammonium oxalate, and ammonium chloride, by action of sodium hypobromite, are given in Table III.

I wish to thank Prof. F. A. Gooch for his advice and kind assistance.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, January 18th, 1906.

Prof. R. MELDOLA, F.R.S., President, in the Chair.

Of the following papers, those marked * were read:—

*1. "*The Refractive Indices of Crystallising Solutions, with especial reference to the Passage from the Metastable to the Labile Condition.*" By HENRY ALEXANDER MIERS and FLORENCE ISAAC.

The authors found that the refractive index of a strong solution of sodium nitrate, measured at intervals while the liquid cools, rises to a maximum value and then falls, crystals appearing before the maximum is reached. If the solution is stirred during the process, the fall is very rapid, and is accompanied by a profuse shower of crystals. Other solutions (for example, alum, sodium chlorate, sodium thiosulphate, ammonium oxalate) behave in the same way. There are always two periods of crystallisation: a first, in which a few crystals are growing gradually; a second, in which many crystals appear spontaneously; so that in the latter the increase of index due to cooling is more than compensated by the decrease due to weakening of the solution. The authors regard these as being undoubtedly the metastable and labile states. This view is confirmed by the behaviour of the same solutions enclosed in sealed tubes, and shaken continuously; if the salt is completely dissolved, they cannot be made to crystallise until they have assumed a temperature corresponding to the maximum refractive index. By means of auxiliary experiments, the refractive indices are interpreted as concentrations, and each series of observations is expressed as a curve with concentrations for ordinates and indices for abscissæ; the maximum points of the curve lie on a new curve, the "supersolubility curve" denoting the limit between the metastable and labile conditions; the same curve is given by the temperatures at which different solutions yield crystals when shaken in sealed tubes; it shows the highest temperature at which any given supersaturated solution can crystallise spontaneously. It is necessary in the case of sodium chlorate to employ friction as well as motion, by enclosing some insoluble substance in the tube, in order to make the crystals appear as soon as the supersolubility curve is reached.

In the experiments in open vessels, the refractive index is measured by means of a totally reflecting glass prism immersed in the liquid; the crystals which appear before the labile state is reached are doubtless introduced with the dust of the air, or are due to evaporation and cooling at the edges and surface of the liquid, which reduces the solution locally to the labile state.

DISCUSSION.

Dr. TUTTON said that it appeared to him that these interesting experiments lead to two important results. Firstly, that determinations of the refractive index of highly concentrated solutions afford valuable indications of the limits of the so-called "labile" and "metastable" conditions, and a new means of exploring the region of supersaturation. Secondly, that this new method of research may be of value in cases of dimorphism, where one of the

forms only is commonly obtained, by throwing some light on the reasons for the predisposition to crystallise in that particular form rather than in the other.

Sir WILLIAM RAMSAY asked whether Professor Miers had made any experiments on the condition of water cooled to below 4° from a high temperature, and produced from melted ice, but not allowed to rise above 4° . Experiments made by Mr. Wilmore in his laboratory had failed to show any difference in density of different specimens of water thus prepared; but he understood that Dr. Chichester Bell had been successful in detecting different values for γ , the ratio of the specific heats at constant volume and constant pressure, in two such specimens of water; γ was determined by an acoustical method.

Mr. PICKERING said that the authors should be congratulated on having obtained a method of studying solutions at that critical period of their existence when crystallisation is occurring, and he suggested that it might be of advantage to study pure liquids in the same way.

In reply to a question from Prof. ARMSTRONG, Prof. MIERS said that the experiments reveal a state of the solution in which a sudden access of spontaneous crystallisation occurs, and this condition is indicated by the term "labile." He hoped to extend the experiments to water and other pure substances.

*2. "The Effect of Constitution on the Rotatory Power of Optically Active Nitrogen Compounds." (Part I.). By MARY BEATRICE THOMAS and HUMPHREY OWEN JONES.

The resolution of a set of optically active nitrogen compounds and the examination of the rotatory power of their salts in dilute aqueous solution have been made in order to find the rotatory power of the ions. This property of the ions is free from many of the disturbing influences such as molecular association and nature of solvent, which affect the rotatory power of non-electrolytes, and render comparable results so difficult to obtain with this class of compound.

Relations between rotatory power and constitution ought therefore to become more evident in the case of ions than of complete molecules.

Two homologous series, each consisting of five compounds, were chosen for examination; in one series, three of the hydrogen atoms in the ammonium ion are replaced by the phenyl, methyl, and allyl groups, whilst the fourth atom of hydrogen is replaced successively by the ethyl, *n*-propyl, isopropyl, isobutyl, and isoamyl groups; in the other series, the phenyl, methyl, and benzyl groups are present with the same five aliphatic groups. The ethyl compound of the latter series has already been described by one of us, and Wedekind has resolved the *n*-propyl and isobutyl compounds. The compounds originally resolved by Pope and Peachey, namely, the phenylbenzylmethylallylammonium salts, fall naturally into both these series.

The following are the values of $[M]_D$ for the ions at 15° :—

Phenylmethylallyl series. Ethyl, 16° ; *n*-propyl, 106° ; isopropyl, 103° ; isobutyl, 55° ; isoamyl, 18° ; benzyl, 167° .

Phenylmethylbenzyl series. Ethyl, 19.4° ; *n*-propyl, 299° ; isopropyl, 398° ; isobutyl, 323° ; isoamyl, 287° .

In all cases examined, the value of $[M]_D$ for the basic ion is diminished slightly with increasing temperature between 10° and 50° .

In both series there is a well-marked maximum of rotatory power at the second member of the series, the propyl or isopropyl compound; in the allyl series, the rotatory power of these two compounds is almost identical, but in the benzyl series the propyl compound occupies quite a different position.

The application of Guye's hypothesis to the case of nitrogen gives an expression for the rotatory power of the compound in terms of the masses of the substituting groups which does not account for the results in an entirely satisfactory manner.

In those cases where the active iodide could be recovered its rotatory power was examined in solution in alcohol and in chloroform.

The rotatory power was always greater in chloroform than in alcohol, and the iodides invariably underwent racemisation in chloroform solution with very different velocities.

*3. "The Determination of Available Plant Food in Soil by the Use of Weak Acid Solvents." By ARTHUR DANIEL HALL and ARTHUR AMOS.

The authors have investigated the effect of repeating the attack of weak acid solvents on soils of known history derived from the Rothamsted experimental plots. After the first extraction, the soil is washed, and again extracted with the same solvent, the process being repeated several times. The solvents employed were water charged with carbon dioxide and a 1 per cent solution of citric acid.

The first extraction does not remove the whole of the phosphoric acid capable of going into solution in the given solvent, the reaction is a reversible one, and a position of equilibrium is attained between the phosphoric acid in solution and the bases in the soil.

With carbon dioxide and water, the position of equilibrium is approximately constant for successive extractions and is characteristic of the manurial history of the soil. With dilute citric acid, the phosphoric acid going into solution falls for the first four or five extractions, and then becomes approximately constant. In the case of the Rothamsted soils, which have been continuously manured with superphosphate, the phosphoric acid going into solution decreases logarithmically, indicating that the amount dissolved is proportional to the active mass of a particular compound present in the soil, six-tenths of which become dissolved at each extraction. The total quantity of this compound is equal to the phosphoric acid supplied as manure during the last fifty years, less that which has been removed in the crop.

Soils which have received more varied compounds of phosphoric acid as manure do not show the same regular decrement in the amounts passing into solution, indicating the presence in the soil of a number of compounds of phosphoric acid of differing solubility.

The investigation lends no support to the theory that all soils establish in the soil water a solution of phosphoric acid of approximately the same composition and independent of the fertilisers the soil receives.

DISCUSSION.

Dr. DYER said the paper was one of much interest; for whilst in his original examination of these soils he had, by one extraction only with citric acid, found in the surface soil the greater part of the calculated accumulation of phosphoric acid due to the manuring, the authors had now, by their supplementary extractions with the same reagent, discovered the remainder almost with the accuracy of an accountant's balance sheet—except in the case of the dunged plots. In three cases, the earlier investigation showed that some of the phosphoric acid had gone down into the lower depths of the sub-soil, and, furthermore, the actual quantity of phosphoric acid added in the dung could only be vaguely guessed. It was satisfactory to hear that the authors still considered that for practical analytical purposes one extraction sufficed.

Mr. PICKERING asked whether the influence of the volume of solution used—which would mean a variation in the proportions of citric acid to soil—had been investigated, also whether the effect of pulverisation of the soil had been examined, for he had found that the amount of potash and iron dissolved by citric acid from a soil depended largely on the degree of fineness to which the soil had been reduced.

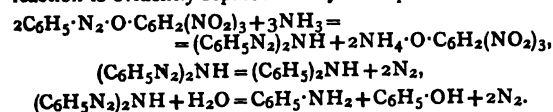
In reply, Mr. HALL said that in all cases the amount of citric acid solution had been kept constant, 1 litre to 100 grms. of the soil. Soil which passed the 3 m.m. sieve was used without any grinding, but as there were other reasons for supposing that the soil phosphates were almost wholly found on the outside of the soil particles, grinding should not make much difference to the phosphoric acid results.

*4. "The Action of Ammonia and Amines on Diazo-benzene Picrate." By OSWALD SILBERRAD and GODFREY ROTTER.

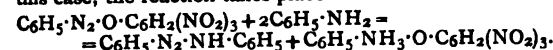
When diazobenzene picrate is acted on by ammonia gas, it rapidly loses the characteristics of a diazo-salt, and forms a dark red product.

Two possibilities presented themselves, namely:—(1) The formation of ammonium picrate, accompanied by further reactions between the diazo-complex and the excess of ammonia; (2) an intramolecular transformation of the diazo-picrate to trinitrobenzeneazophenol, somewhat analogous to the diazoaminobenzene transformation.

The intense red colour of the product appeared to lend some probability to the latter assumption, but an examination of the reaction completely disproved the presence of azo-compounds. The products proved to consist mainly of ammonium picrate, and diphenylamine, aniline, and phenol were also detected. Thus, the course of the reaction is evidently represented by the equations—



Other compounds occurred in small quantities as the result of secondary reactions. When treated with aniline, diazobenzene picrate gives rise to aniline picrate and aminoazobenzene; the latter compound evidently resulted from a transformation of diazoaminobenzene; thus, in this case, the reaction takes place as follows:—



DISCUSSION.

Dr. MORGAN stated that in conjunction with Mr. Wootton he had studied the picrates of diazo-compounds, and especially the stable product derived from *p*-amino-benzanilide (*Proc.*, xxii., p. 23). This compound was precipitated even in strongly acid solution, and was therefore in all probability a diazonium derivative. They had not detected *syn*- and *anti*-isomerism in this series. It would be interesting to know whether the more explosive diazo-benzene picrate was a diazo-compound or a diazonium salt.

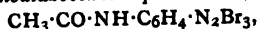
In reply, Dr. SILBERRAD said that the comparative stability of diazobenzene picrate was remarkable considering its constitution, but, nevertheless, he regarded it as a true diazonium salt. Under these conditions, it seemed improbable that *syn*- and *anti*-derivatives could be prepared, which was in accord with Dr. Morgan's observations on similar compounds.

*5. "The Preparation of Bistriazobenzene." By OSWALD SILBERRAD and BERTRAM JAMES SMART.

The explosive properties of bistriazobenzene have hitherto precluded the verification of its composition by analysis; for this reason, a re-investigation of the compound has been undertaken, and at the same time the method for its preparation has been somewhat modified.

Griess (*Ber.*, 1888, xxi., 1559) took the oxamic acid of *p*-phenylenediamine as the starting-point for the preparation of this compound; in the present work, however, it has been found advantageous to proceed from *p*-amino-acetanilide. The successive steps of the preparation are seen from the following series of intermediate products:—*p*-Aminoacetanilide, acetyl-*p*-aminodiazobenzene perbromide, *p*-aminotriazobenzene, *p*-triazodiazobenzene perbromide, bistriazobenzene.

Acetyl-*p*-aminodiazobenzene perbromide,—



crystallises in small yellow plates (m. p. 126°) with decomposition.

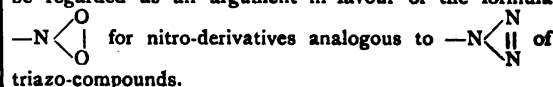
p-Aminotriazobenzene, $\text{NH}_2\cdot\text{C}_6\text{H}_4\cdot\text{N}_3$, was prepared by the hydrolysis of acetyl-*p*-aminotriazobenzene with caustic potash, previously unsuccessfully attempted by Rupe and von Majewski (*Ber.*, 1900, xxxiii., 3406).

Bistriazobenzene, $\text{N}_3\cdot\text{C}_6\text{H}_4\cdot\text{N}_3$ (m. p. 83°) explodes on heating; its analysis was, however, successfully carried out under diminished pressure, and gave results which agreed with the above formula.

DISCUSSION.

Dr. FORSTER inquired whether *p*-bistriazobenzene has a distinctive odour, because he had noticed that *m*- and *p*-nitrophenylazoimides have a faint odour of anise, which is more marked in the case of *p*-tolylazoimide and *p*-chlorophenylazoimide, whilst in the case of *p*-methoxyphenylazoimide the odour is comparable with that of anethole itself, being less sweet and more pungent than the perfume of the last-named substance. Curiously enough, *o*-nitrophenylazoimide is odourless.

In reply, Dr. SILBERRAD said that he had noticed an anise-like odour, but considered the smell more similar to that of a nitro-compound, and suggested that this might be regarded as an argument in favour of the formula



*6. "Gradual Decomposition of Ethyl Diazoacetate." By OSWALD SILBERRAD and CHARLES SMART ROY.

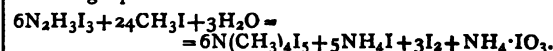
When ethyl diazoacetate is exposed to light at the ordinary temperature, a gradual evolution of nitrogen occurs, and after the space of several years, thick colourless rhombic crystals resembling cane-sugar gradually make their appearance. On examination, this compound proved to be the triethyl ester of 4:5-dihydro-3:4:5-pyrazoletricarboxylic acid.

Subsequent work showed that the same compound can readily be produced by warming ethyl diazoacetate with copper dust. In both cases, the reaction is evidently due to the intermediate formation of fumaric or maleic esters.

This view is confirmed by the work of Buchner and Witter, who prepared the trimethyl ester by the action of the corresponding ester of fumaric or maleic acid on methyl diazoacetate (*Annalen*, 1895, cclxxiii., 239).

*7. "Studies on Nitrogen Iodide. III. The Action of Methyl and Benzyl Iodides." By OSWALD SILBERRAD and BERTRAM JAMES SMART.

The Action of Methyl Iodide.—Previous experiments by Stahlschmidt (*Poggendorff's Ann.*, 1863, cxix., 421) on the interaction of nitrogen iodide with methyl iodide led him to represent the reaction by the following equation:— $2\text{NI}_3 + 6\text{CH}_3\text{I} = \text{N}(\text{CH}_3)_4\text{I}_5 + \text{NH}_4\text{I} + 2\text{CHI}_3$. Since the formula here assumed for nitrogen iodide is not in accord with that proved by Chattaway (*Proc.*, 1899, xvii., 20; *Am. Chem. Soc.*, 1900, xxiii., 363, &c.), or with the constitution $\text{NI}_3 : \text{NH}_3$, established by one of the authors (Silberrad, *Trans.*, 1905, lxxxvi., 55 and 66), renewed experiments have been carried out on the above reaction. The formation of tetramethylammonium penta-iodide as observed by Stahlschmidt was confirmed in the present work, and its nature established by its preparation from tetramethylammonium iodide and iodine. Iodoform was not, however, as he believed, a product of the reaction between nitrogen iodide and methyl iodide, but resulted from the alcohol present in his experiments. In the present work, alcohol was excluded; the nitrogen iodide was prepared from ammonia and iodine chlorine, and was allowed to react with excess of methyl iodide under water. The products found were tetramethylammonium penta-iodide, ammonium iodide and iodate, and free iodine. The quantities of all these products were determined, and the reaction was proved to proceed almost quantitatively according to the following equation:—



No compounds corresponding to Stahlschmidt's di-iodo-methylamine or butyric acid were produced.

Action of Benzyl Iodide.—This iodide was found to react readily with moist nitrogen iodide. The nature of

products, however, depends on the proportion of benzyl iodide taken, bright green or garnet-red crystals being deposited according to the conditions of the experiment. These proved both to be periodides of tribenzylamine.

Tribenzylammonium Pentaiodide, $N(C_7H_7)_3HI_5$, which forms bright green crystals melting at $121-122^\circ$, is sparingly soluble in alcohol, and undergoes a slight decomposition on re-crystallisation.

Tribenzylammonium Di-iodide, $N(C_7H_7)_3HI_2$, crystallises in garnet-red prisms melting at $115-116^\circ$; it dissolves more readily in alcohol than the pentaiodide, and can be more easily obtained in the crystalline condition.

The pentaiodide can be readily converted to the red di-iodide by the partial abstraction of its iodine by shaking with potassium iodide solution. Conversely, the di-iodide can be converted into pentaiodide by treatment with alcoholic iodine. Both compounds can also be obtained direct from tribenzylamine by the addition of the required quantity of iodine to the iodide of the base.

*8. "Action of Bromine on Benzeneazo-o-nitrophenol." By JOHN THEODORE HEWITT and NORMAN WALKER.

By the action of bromine on 4-benzeneazo-2-nitrophenol in presence of glacial acetic acid and fused sodium acetate, the 6-hydrogen atom is replaced. The constitution of the resulting compound, which when pure melts at $154.5-155^\circ$, was confirmed by fission with excess of fuming nitric acid. The resulting *p*-nitrophenyldiazonium nitrate was isolated by conversion into *p*-nitrobenzeneazo- β -naphthol, whilst 1.5 grms. of 2-bromo-4:6-dinitrophenol, melting before purification at 116° (uncorr.), were obtained instead of the theoretical amount of 1.6 grms. from 2 grms. of the azophenol. Körner gives the melting-point of bromodinitrophenol at 118° .

The following derivatives of benzeneazobromonitrophenol have been analysed:—Sodium and potassium salts, acetyl derivative (m. p. 137°), and benzoyl derivative (m. p. 131°). 4-*p*-Tolueneazo-2-nitro-6-bromophenol was prepared in a similar manner from *p*-tolueneazobromonitrophenol; it melts at 161° . Fuming nitric acid furnishes 2-bromo-4:6-dinitrophenol as a product of fission; its acetyl derivative melts at 124° , the benzoyl derivative at 129° .

*9. "The Condensation of Dimethyldihydroresorcin and of Chloroketodimethyltetrahydrobenzene with Primary Amines. Part I. Monoamines, Ammonia, Aniline, and *p*-Toluidine." By PAUL HAAS.

Dimethyldihydroresorcin reacts with ketonic reagents as a diketone; in most of its other reactions, however, it behaves as a hydroxyketone having the formula $CM_2 < \begin{smallmatrix} CH_2 & C(OH) \\ & | \\ & CO \end{smallmatrix} \equiv CH$. In this form, it condenses with a single molecule of a primary amine by the elimination of water between the hydroxyl group and the amino-hydrogen, giving rise to a secondary amine. This replacement of the hydroxyl group by a basic group $-NHR$ caused the remaining ketonic oxygen to become hydroxylic, as shown by the fact that the new substance gives a colour reaction with ferric chloride and does not react with ketonic reagents; when, however, the basic substituting group is rendered more acid by acetylation, the resulting compound no longer gives any coloration with ferric chloride, and condenses with semicarbazide, showing that the oxygen atom has become ketonic.

Chloroketodimethyltetrahydrobenzene, unlike dimethyldihydroresorcin, reacts at once with two molecules of a primary amine, giving rise to the hydrochloride of a mixed secondary and tertiary base having the formula $CM_2 < \begin{smallmatrix} CH_2 & C(NHR) \\ & | \\ & C(NR) \end{smallmatrix} \equiv CH$. Bases of this type may also be prepared by condensing the mono-substituted derivatives, obtained by the action of dimethyldihydroresorcin on the amine, with a second molecule of the same amine in the presence of zinc chloride.

10. "Silicon Researches. Part X. Silicon Thiocyanate." By J. EMERSON REYNOLDS.

The author found that silicon thiocyanate, $Si(SCN)_4$, is

best prepared by prolonged digestion of pure lead thiocyanate in a benzene solution of silicon tetrachloride. An excess of the lead salt must be used, as the latter exchanges but one of its two thiocyanogen groups for chlorine, forming the yellow crystalline compound $PbCl(SCN)$, silicon thiocyanate remaining in solution. The latter compound is obtained in fine white crystals, when most of the benzene is removed by distillation; but if any moisture is present the crystals become yellow owing to decomposition. This method of preparing the thiocyanate gives a much better product than Miquel's (*Ann. Chim., Phys.*, 1877, [v.], xi., 343), in which the materials are heated to about 400° , as in this investigator's process charring results, and an impure substance is obtained.

The pure thiocyanate melts at 143.8° (corr.) and boils at 314.2° (corr.), distilling without decomposition.

The chief interest attaches to the constitution of the compound, as the somewhat analogous phosphorus "trithiocyanate" has been shown by A. E. Dixon (*Trans.*, 1901, lxxix., 541) to exhibit "a form of tautomerism," in some cases acting as $P(NCS)_3$, and in others as $P(SCN)_3$.

The author has compared silicon thiocyanate with the phenylamide, $Si(NH \cdot C_6H_5)_4$, described in Part V. of this series of papers, and has examined several interactions of the thiocyanate with aniline and other substances, and concludes from the results that it is correctly represented by the expression $Si(SCN)_4$.

11. "Halogen Derivatives of Substituted Oxamides." By FREDERICK DANIEL CHATTAWAY and WILLIAM HENRY LEWIS.

The action of the halogens on substituted oxamides has been little studied, and the description given of the substances formed is not very satisfactory, as the crude products obtained were not subjected to any process of purification. The action of chlorine on oxanilide has been studied, and a number of substituted oxanilides has been prepared in a simpler way in order to compare them with the foregoing products.

The usual product of the chlorination of oxanilide is a mixture of *s*-di-*p*-chlorophenyloxamide and of its *s*-dichloro-amino-derivative.

When aniline or a substituted aniline is heated with ethyl oxalate, a symmetrical disubstituted oxamide on the ethyl ester of a substituted oxamic acid is formed according as the aniline or the ethyl oxalate is in excess.

The symmetrical chlorophenyloxamides, which are almost insoluble in ordinary solvents, crystallise well from nitrobenzene, in which on heating they readily dissolve.

The following compounds were described:—*s*-Di-*p*-chlorophenyloxodichloroamide, colourless rhombs (m. p. 169°); ethyl *p*-chlorophenyloxamate, colourless plates (m. p. 155°); *p*-chlorophenyloxamide, colourless needles (m. p. 241°); *s*-di-*p*-chlorophenyloxamide, colourless thin plates (m. p. 288°); ethyl 2:4-dichlorophenyloxamate, colourless, hair-like prisms (m. p. 119°); 2:4-dichlorophenyloxamide, colourless, hair-like crystals (m. p. 234°); *s*-di-2:4-dichlorophenyloxamide, colourless, slender prisms (m. p. 276°); *s*-dimethyloxodichloroamide, colourless, slender prisms (m. p. 37°); *s*-diethyloxodichloroamide, pale yellow, viscid liquid which decomposes on heating; *s*-dimethyloxodibromoamide, pale yellow, flattened prisms (m. p. 95°); *s*-diethyloxodibromoamide, pale yellow plates (m. p. 82°).

12. "Menthyl Benzenesulphonate and Menthyl Naphthalene-6-sulphonate." By THOMAS STEWART PATTERSON and JOHN FREW.

Menthyl benzenesulphonate and menthyl naphthylene- β -sulphonate have been prepared by the pyridine method. The former crystallises from alcohol in silky needles melting at 80° , the latter separates from alcohol in solid, opaque crystals melting at $114-114.5^\circ$. The compounds themselves decompose on continued heating at or above the melting-point, so that their rotations have been determined in ethyl alcohol, benzene, and nitrobenzene. The chief points of interest are as follows.—1. There is con-

siderable variation of rotation with change of solvent. 2. The values of $[M]_D$ for the β -naphthalene compound are consistently lower than for the benzenesulphonate in a given solvent. 3. The solvents, however, do not exert proportionate influences on the rotations of the two compounds. 4. Menthyl benzenesulphonate has a lower molecular rotation than menthyl benzoate by about 56° , and menthyl naphthalene- β -sulphonate a lower rotation than menthyl β -naphthoate by about 113° . 5. As regards the influence of temperature change on the rotations of these compounds in solution, it was found that the greater the rotation produced by a solvent the more rapid was its diminution with rise of temperature. The molecular rotations in dilute solution seem to tend at higher temperatures towards a common value of about -170° .

13. "Some Reactions and New Compounds of Fluorine." By EDMUND BRYDGES RUDHALL PRIDEAUX.

The fluorine was prepared by the electrolysis of anhydrous hydrogen fluoride contained in a copper vessel (Moissan, "Le Fluor et ses Composés"). In every case when the gas was analysed it was found to contain oxygen, and special experiments proved that this oxygen was produced at the anode even after the current had passed for a considerable time.

Liquid fluorine has no solvent or chemical action on iodine. The colourless iodine fluoride was analysed by a volumetric method, and four concordant percentages of iodine confirmed the formula IF_3 established by Moissan (*Comptes Rendus*, 1902, cxxxv., 563) from the results of gravimetric analysis.

Bromine fluoride was prepared for the first time; analyses carried out by a gravimetric method led to the formula BrF_3 . Liquid fluorine has neither solvent nor chemical action on solid bromine.

Gaseous fluorides of selenium and tellurium were prepared by the direct combination of the elements in the cold. The formulæ of these gases were proved to be SeF_6 and TeF_6 by determinations of their densities. Analysis of TeF_6 also showed it to have this formula. These gases are colourless and easily condensable by moderate cold or pressure to white crystalline solids and colourless liquids respectively. Tellurium hexafluoride has an unpleasant odour, and decomposes water slowly at 15° ; selenium hexafluoride does not decompose water at 15° .

The vapour pressure curve of SF_6 was determined for comparison with those of SeF_6 and TeF_6 . The three curves resemble one another closely. In the case of SF_6 and SeF_6 , the melting-points lie above the temperatures at which the vapour over the solid attains a pressure of 760 m.m., whilst in the case of TeF_6 the melting-point lies just below the boiling-point. The critical temperatures of the liquefied gases rise in the order SF_6 , SeF_6 , TeF_6 .

The molecular volumes at temperatures equally removed from the melting-points were found to be very nearly the same.

The refractivities of the three compound gases bear no simple additive relation to those of their constituent elements.

The refractivities minus a constant are directly proportional to the densities of the gases.

(To be continued).

PHYSICAL SOCIETY.

Ordinary Meeting, January 26th, 1906.

Prof. J. H. POYNTING, F.R.S., President, in the Chair.

A PAPER ON "The Isothermal Distillation of Nitrogen and Oxygen and of Argon and Oxygen" was read by Mr. I. K. INGLIS.

Mixtures of liquid nitrogen and oxygen are particularly suitable for an exact study of the relations obtaining during isothermal distillation; for the distillation bulb being the coldest instead of the hottest part of the apparatus, errors

due to back condensation, &c., can be eliminated. In addition the vapour can easily be circulated and passed time after time through the liquid until equilibrium is complete. Experiments were carried out in this way in a specially designed apparatus, and the results showed that the ratio of nitrogen to oxygen in the vapour was not in a constant proportion to the same ratio in the liquid. When, however, the partial pressures of nitrogen and oxygen were plotted against the concentrations in the liquid, a straight line was obtained in the case of nitrogen and a curve in the case of oxygen. This indicated that nitrogen obeyed Henry's law of solubility, and the deviation in the case of oxygen pointed to its being slightly associated in the liquid state when mixed with nitrogen. A few experiments were also carried out with mixtures of argon and oxygen. At the temperature used argon was a volatile solid, and therefore the greatest concentration of argon that could be obtained was that of the saturated solution in oxygen. Argon seemed to agree with nitrogen in obeying Henry's law.

Dr. J. A. HARKER said the paper was interesting at the present time, as in the *Journal de Physique* for January a new process for obtaining liquid oxygen commercially and with the expenditure of a little power was described, in which advantage was taken of the difference in composition of the liquid and the vapour in the distillation of liquid air.

A paper on "The Use of Chilled Cast-iron for Permanent Magnets" was read by Mr. A. CAMPBELL.

The experiments described were undertaken with a view to corroborating and extending the results already obtained by other experimenters. A good many years ago Mr. J. R. Ashworth showed that rods of chilled cast-iron could be made into very fair permanent magnets; and last year Mr. B. O. Peirce, of Harvard University, published an interesting research on the subject, going considerably farther, and describing the practical utility of chilled cast-iron for magnets of moving-coil galvanometers and other instruments. His tests, although conclusive in many ways, did not in general give any absolute values (for permeability, &c.). The present investigation was made on rings in addition to rods of the standard dimensions usual in testing magnet-steels. All the specimens were heated to $1000^\circ C.$, and then chilled in cold water, care being taken to support them during the process, as cast-iron is very brittle at such a high temperature. The rods were tested for maximum remanent B and coercivity by Madame Curie's method, the magnetised bar being placed in a long solenoid producing a demagnetising field which was gradually increased until a search-coil slipped along the bar showed that the demagnetisation was complete. The results showed the chilled cast-iron to be not very inferior to ordinary magnet steel. By ballistic tests on the two rings, their permeability curves were obtained, and these indicated that the simple process of chilling used was quite satisfactory even for a tolerably massive ring of 6 sq. c.m. cross section. The cheapness and ease of working cast-iron should encourage instrument-makers to test its capabilities in various instruments.

Mr. W. H. PRETTY asked the author what kind of cast-iron he had used in his experiments. The physical properties of cast-iron varied with the chemical composition, and it was important that the kind of iron used in the experiments should be stated.

The AUTHOR, in reply to Mr. Pretty, said the iron used was grey commercial cast-iron, but Mr. Ashworth had tested several kinds and found very little difference in their magnetic properties.

A paper by Prof. LYLE and Mr. BALDWIN, "On Experiments on the Propagation of Longitudinal Waves of Magnetic Flux along Iron Wires and Rods," was read by Prof. F. T. TROUTON.

The experiments described in the paper were undertaken with the object of determining if there is a definite rate of propagation of magnetism in iron. The method adopted

was to produce magnetisation at a particular point on a bar by means of a coil through which an alternating current was passed, and then to observe the magnetic flux at various distances from the coil by means of a small secondary coil, free to be moved to various places on the bar. By the use of Prof. Lyle's wave-tracer the magnetic flux at various points along the bar was thus obtainable. The wave curl was then analysed by Fourier's series. Various curves given in the paper show the value of the constants in Fourier's series, and of the lag in the magnetisation as the coil was moved along the bar. Contrary to what had been observed in previous researches, the authors found that the phase lag, instead of continuously increasing along the bar, reached a maximum value and then diminished, proving the absence of true wave propagation.

Prof. TROUTON read a letter from Mr. A. W. PORTER pointing out that the occurrence of a maximum in the phase lag was exactly what was to be expected on any theory which made the lag depend upon a direct power of both permeability and distance.

CORRESPONDENCE

THE TEMPERATURE OF SOLUTIONS HEATED BY OPEN STEAM.

To the Editor of the Chemical News.

SIR,—Allow me to correct the erroneous impression—conveyed by Mr. Steel's article on the above subject (CHEMICAL NEWS, vol. xciii., p. 42)—that, although Peter Spence discovered that open steam would heat solutions to higher temperatures than its own, and described his experiments in detail in the *Brit. Assoc. Trans.* for 1869, he had no explanation to give of the phenomenon; and that the first person to theoretically account for it was H. Claassen, thirty-five years after (*i.e.*, in 1904).

The simple fact is: the explanation has been familiar at these works and elsewhere ever since 1869.

I append in evidence of this the subjoined extract from p. 31 of the *Brit. Mer. Gazette* for July, 1878.—I am, &c.,

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"Some very important scientific discoveries have been made by Mr. Spence, apart from those more directly affecting his own branch of the chemical industry, whilst prosecuting his experiments and studies. He ascertained that steam of 212° F. could be made to raise the temperature of saline solutions to their boiling-point, however high that boiling-point might be. Thus a solution of acetate of potash might be readily raised by open steam to a temperature of 336° F. This result appeared so paradoxical that on Mr. Spence reading a paper on the subject, at the Exeter Meeting of the British Association, his statements were received with incredulity until the actual experiment was successfully made before his auditory, which included Prof. Williamson and other able chemists. The fact being proved, the explanation, as in the case of Columbus and the egg, was readily received, *viz.*, that the latent heat evolved by the condensing steam became sensible heat, measurable by the thermometer."

COLOUR AND STRUCTURE.

To the Editor of the Chemical News.

SIR,—The relation between constitution and physical properties has been a matter of the deepest interest to chemists for many years, but your readers may not be

aware that it is beginning to attract the sympathetic attention of the British working man.

At this end of the General Election everybody must be familiar with the illustrated Anti-Tea Duty poster, entitled "It Don't seem Right to Me." On my way to the meeting of the Chemical Society last Thursday, when there was some discussion on the above subject, a pamphlet was placed in my hands, also entitled "It Don't seem Right to Me." The pamphlet is in the following terms:—

"Talk of no colour without quinine;
Well, I'll tell you what I think:
There's many a compound isn't blue,
What's neither green nor pink.
No, neither pink nor green they are,
And it don't seem right to me,
To say that a compound ain't a quinine
When the N's double-linked with C."

I enclose my card, and subscribe myself,—Yours, &c.,

ETHENOID LINKAGE.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances de l'Académie des Sciences. Vol. cxlii., No. 2, January 8, 1906.

New Type of Compound in the Rare Earth Group of Metals.—C. Matignon and E. Cazes.—Anhydrous samarium chloride, SmCl_3 , has the property of being reduced by hydrogen, giving rise to a sub-chloride. This reduction necessitates a high temperature, and is best performed in a thick tube of Jena glass. The substance blackens when the reduction starts, and then melts and apparently boils, owing to the evolution of hydrochloric acid gas. The reaction is reversible, and can be expressed by the following equation:— $\text{SmCl}_3 + \frac{1}{2}\text{H}_2 \rightleftharpoons \text{SmCl}_2 + \text{HCl}$. The reduction can be better effected by using ammonia gas:— $3\text{SmCl}_3 + 4\text{NH}_3 = 3\text{SmCl}_2 + 3\text{NH}_4\text{Cl} + \text{N}$. It seems probable that praseodymium and neodymium are susceptible of also forming a sub-chloride under suitable conditions.

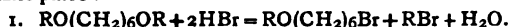
Electrolytic Preparation of Spongy Tin.—D. Tommasi.—The apparatus which may be used for the preparation of spongy tin consists of a rectangular vessel containing a solution consisting of 50 parts of water, 10 parts of stannous chloride, and 1 part of hydrochloric acid. Into this bath two tin anodes are placed, between which is a cathode consisting of a copper disc fixed by its centre in such a manner as to be capable of rotation, so that half the disc is always in contact with the air and half with the liquid.

Cuprous Silicide.—Em. Vigouroux.—The author confirms a set of experiments which he performed in 1901 on pure copper silicides, in which he finds the proportion of combined silicon very nearly 10 per cent. He also now isolates cuprous silicide, Cu_4Si , and examines its principal properties.

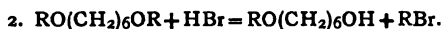
Reduction of Silver and Copper Chlorides by means of Calcium.—L. Hackspill.—By heating in a porcelain vacuum tube an iron boat containing variable proportions of silver chloride and calcium, the author obtains alloys in which the proportion of calcium can be as great as 45 per cent:— $2\text{AgCl} + n\text{Ca} = \text{CaCl}_2 + (n-1)\text{Ca} + 2\text{Ag}$. The above reaction takes place at a dull red heat. An examination of the silver and calcium alloys shows them to have a totally different appearance from silver; they are grey, crystalline, and can easily be powdered. When heated in the air they rapidly oxidise. After being heated for some time in air all the calcium is transformed into lime, which floats on the surface of the silver. All the alloys are attacked by cold water.

Asymmetric Derivatives of Hexanediol-1.6-diethyl Ether and Heptanediol-1.7-diiodide.—

R. Dionneau.—When hydrobromic acid acts on the ether oxides of hexanediol-1.6 in the cold, the following reaction takes place :—



This reaction is most complex. On the side of the non-transformed ether are bodies of alcoholic function, amongst which hexamethylenic glycol has been isolated and recognised by its fusing-point. Besides Reaction 1, which produces the bromide function on the hexamethylenic group, there is a secondary reaction which gives rise to the alcoholic function :—



Certain Derivatives of the Fatty Carbides produced by Hydrogenation by means of Ammonium Metals. Preparation of Ethylenic and Formenic Carbides.—E. Chablay.—The author finds that the molecule of an alkaline ammonium metal reacts as a hydrogenating agent on the bi-substituted halogen derivatives, and also on tri-substituted derivatives, with the condition that all substitutions must be made on the same atom of carbon. Such a molecule, on the contrary, reacts simply by virtue of its alkaline metal when the two substitutions are made on different carbons. The example of trimethylene bromide seems to indicate that the reactions have a specially simple character when the double substitution is made on two neighbouring atoms of carbon.

MISCELLANEOUS.

Royal Institution.—On Thursday next, February 15, at 5 o'clock, Mr. H. B. Irving delivers the first of two lectures at the Royal Institution on "The English Stage in the Eighteenth Century"; and on Saturday, February 17, at 3 o'clock, Mr. M. H. Spielmann begins a course of two lectures on "George Frederick Watts as a Portrait Painter." The Friday Evening Discourse on February 16 will be delivered by Mr. W. C. D. Whetham on "The Passage of Electricity through Liquids," and on February 23 by Professor J. O. Arnold on "The Internal Architecture of Metals."

Relative Vapour Tensions of the Three Different Modifications of Carbon.—A. Smits.—Rudolf Schenck and W. Heller (*Berichte*, 1905, xxxviii., 2139; *CHEMICAL NEWS*, 1905, xcii., 83) have shown that the different modifications of carbon possess different amounts of free energy, and that the equilibrium constants between CO, CO₂, and the different forms of solid carbon must have different values at the same temperature, being greatest for the most labile form and least for the most stable. But the authors now point out that, though these statements are quite correct, the phenomena may be regarded in a different light, the difference in the values of the equilibrium constants being due to the different vapour tensions of the modifications of carbon at the same temperature. From the equilibrium equations it appears that the equilibrium pressures are proportional to the vapour tensions of the different varieties. If we imagine a closed exhausted space containing diamond and graphite, both forms would emit carbon vapour, the diamond more than graphite because its vapour tension is greater. It would then be expected that as soon as the vapour tension had exceeded that of graphite the carbon vapour would condense to graphite, which is not the case. Therefore we must assume that the carbon pressure rises to the vapour pressure of diamond. The vapour is supersaturated as regards graphite, and yet no condensation takes place. This phenomenon probably often occurs in the case of substances with very low vapour tensions. As regards the system consisting of ferrous oxide and one of the meta-stable forms of carbon, Schenck and Heller have shown that the carbon which is formed from carbon monoxide in contact with iron is graphite. Thus while carbon dioxide

is reduced by diamond carbon vapour to the monoxide, the latter yields graphite instead of diamond, and the meta-stable form disappears. The author has come to the conclusion that Schenck and Heller in their experiments with amorphous carbon and diamond did not measure the equilibrium pressures, and that if they had been able to continue their experiments they must have observed a decrease of the equilibrium pressure with amorphous carbon and diamond, until that pressure had been reached which corresponds to graphite. From their experiments it appears that although the absolute magnitude of the vapour tension of carbon is so small it is quite appreciable in equilibrium work of this kind.—*Berichte*, xxxviii., No. 17.

MEETINGS FOR THE WEEK.

- MONDAY, 12th.—Society of Arts, 8. (Cantor Lectures). "Modern Warships," by Sir William White, F.R.S., &c.
TUESDAY, 13th.—Royal Institution, 5. "Food and Nutrition," by Prof. William Stirling, M.D., &c.
WEDNESDAY, 14th.—Society of Arts, 8. "The Horseless Carriage, 1885-1905," by Claude Johnson.
THURSDAY, 15th.—Royal Institution, 5. "The English Stage in the Eighteenth Century," by H. B. Irving, M.A.
Society of Arts, 4.30. "The Navigable Waterways of India," by Robert B. Buckley, C.S.I.
Chemical, 8.30. "Cuprous Formate," by A. Angel.
"Solubility of Triphenylmethane in Organic Liquids with which it forms Crystalline Compounds," by H. Hartley and N. G. Thomas.
"Spontaneous Crystallisation of Supersaturated Solutions," by H. Hartley.
"Preparation and Properties of some New Tropines," by H. A. D. Jowett and A. C. O. Hann.
"Studies in Asymmetric Synthesis—Part IV. The Application of Grignard's Reaction for Asymmetric Syntheses," by A. McKenzie.
FRIDAY, 16th.—Royal Institution, 9. "The Passage of Electricity through Liquids," by W. C. D. Whetham, F.R.S.
SATURDAY, 17th.—Royal Institution, 3. "George Frederick Watts, as a Portrait Painter," by M. H. Spielmann.

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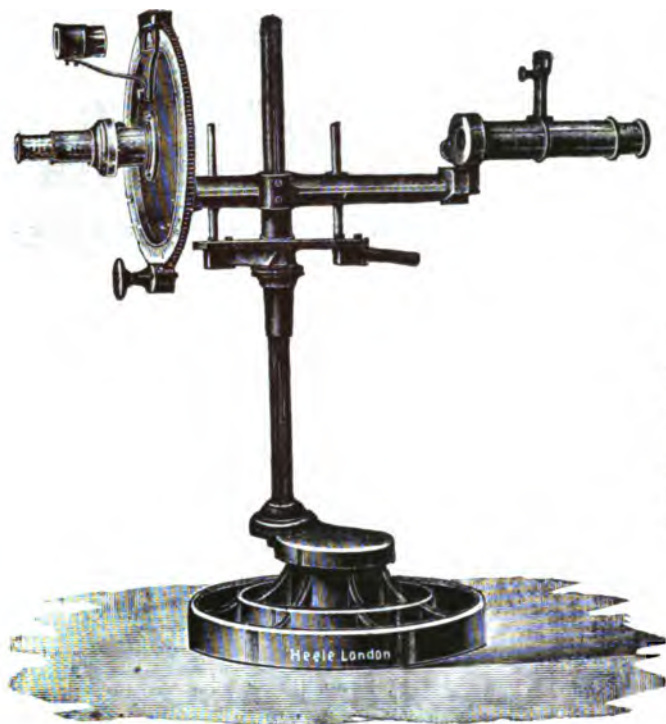
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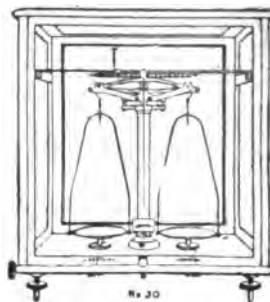
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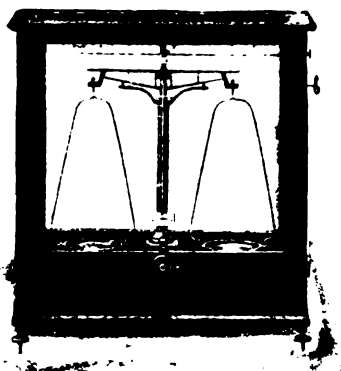
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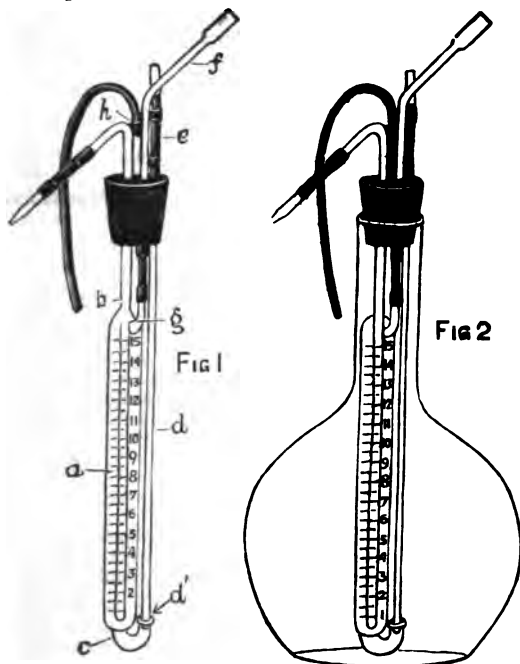
THE CHEMICAL NEWS

VOL. XCIII., No. 2412.

NOTE ON A COMBINED WASH-BOTTLE AND PIPETTE.*

By J. W. HOGARTH.

DURING some chemical work in which it was desirable to dissolve a precipitate, with which the work was concerned, in a known volume of acid, the want was felt of a vessel possessing the combined advantages of a measuring vessel and wash-bottle. It was not originally intended to have devoted a separate article to the description of the apparatus which is described below, but because the work for the time being has been stopped, it was deemed advisable to now make mention of the vessel. Ordinarily when it is wished to dissolve or wash a precipitate with a known volume of solution, a measured quantity of the hot or cold liquid is poured into the filter containing the substance to be dissolved or washed, in which manner it is not possible to stir up the precipitate and thereby offer as large a surface as possible to the action of the solution, consequently



more liquid has to be used than is necessary to bring about the desired change; this excess of solution in some cases may be objectionable. If the liquid be delivered from a wash-bottle the objections to a great extent are obviated, but the volume of liquid used cannot conveniently be arrived at, especially when the filtrate has to be quantitatively treated, because any extra operation tends to introduce errors into the analysis. The apparatus of which the following is a description was designed to overcome the above objections.

The measurements given are those of the actual apparatus made for use. Vessel *a*, which is of thin glass 13.3 c.m. long, and 1.4 c.m. wide, is graduated in cubic centimetres

and fractions thereof. Tube *b* is 5 m.m. in diameter and 13.3 c.m. long from junction at top of *a* to the bottom where it just enters *c* (to bring *b* out more clearly for the purpose of photographing, it was partly filled with coloured liquid). Tube *g* is sealed on to *a*, and is connected to the lower extremity of *f* by a piece of stout rubber tubing. To use the vessel, rod *d* is slightly raised, the water-tight joint at *d'* where rod *d* is ground into *c*, is thereby broken, air is blown from the mouth through the rubber connected to *h*, which is in direct communication with the air of flask only, until the level of liquid rises to the desired mark in *a*; rod *d* is then released, when it is automatically forced back into position by the rubber *e*, which is stretched from the disc on *d* to the out-turned edge of a short glass tube through which *d* passes. This liquid is then delivered in the ordinary way by blowing into *f*; as soon as the liquid in *a* falls to the curved tube *c* liquid ceases to pass up *b*.

The vessel, which was of 15 c.c. capacity, delivered any desired volume within that limit to $\pm \frac{1}{2}$ c.c. Fig. 2 shows the apparatus as it fits in ordinary flask. As far as can be ascertained this apparatus has not elsewhere been described.

The author here takes the opportunity of tendering his thanks to Acting Professor Schofield for the interest shown and encouragement that he has often given him.

A BURETTE-FILLING DEVICE.

By EDWARD FRENCH.

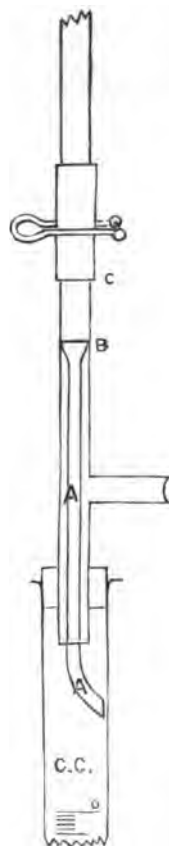
EVERY one who has worked with burettes having side tubes and stopcocks for filling the instrument appreciates the advantages gained by such a device over the old way of filling, by means of a funnel, from the bottle containing the standard solution.

In many laboratories, however, where there is a set of ordinary burettes in daily use, the cost of the new instruments properly fitted prohibits their introduction, and much time is lost in consequence.

By means of the little apparatus about to be described ordinary burettes may be rendered self-filling, and thus a burette which has been calibrated and in actual use does not require to be discarded.

It consists, as will be seen from the annexed figure, of a T-tube of such a diameter as will enable it to be fixed by means of a cork (or indiarubber stopper) to the burette. Passing down the T-tube is a narrow tube *A* made by suitably drawing out a piece of wider tube. At *B* this tube *A* is fused to the T-tube, or it may be fixed by means of rubber. The narrow tube is then bent and cut off so that the end just touches the wall of the burette. Connection is made with the syphon tube from the stock bottle by means of a rubber tube at *c*.

This tube is provided with a pinch-cock, which, when open, allows the standard solution to flow down the walls of the burette, air bubbles being thus avoided. The displaced air escapes by the side branch of the T, and when the burette is in use air enters at the same place. When the work is finished for the time being, this tube is closed by a piece of rubber carrying a glass plug, or by a small rubber teat.



* From the *Journal and Proceedings of the Royal Society of N. S. Wales*, xxxviii. p. 418.

The apparatus is being made by Messrs. Baird and Tatlock, 45, Renfrew Street, Glasgow, who send it out with the narrow tube straight so that the purchaser may bend it to suit a particular burette.

Chemical Laboratory,
The Technical College, Paisley, N.B.

SOLUTION: FRACTIONAL EXTRACTION.

By PHILIP BLACKMAN.

If a substance is soluble in two liquids which are themselves immiscible, then more of the substance by weight dissolved in one of the liquids can be extracted from the solution by using the other liquid in portions than by employing it all in one extracting operation. The molecular weight of the substance is supposed to be the same in both liquids.

Let W = weight of the dissolved substance,
 v_1, v_2 = volumes of the extracting liquid and of the solvent respectively,
 s_1, s_2 = coefficient of solubility of the substance in the respective liquids.

1. If the operation of extraction be performed by using the total volume, v_1 , of the liquid, then, if w = weight of solute extracted, $W - w$ = weight of solute remaining, and $w/(W - w) = v_1 s_1 / v_2 s_2$ or $w + w \cdot v_1 s_1 / v_2 s_2 = W \cdot v_1 s_1 / v_2 s_2$. Hence—

$$w = \frac{v_1 s_1}{v_2 s_2} W / \left(1 + \frac{v_1 s_1}{v_2 s_2} \right) \dots \dots \dots 1.$$

2. If the extracting process be performed in n operations, using each time the volume v_1/n of the extracting liquid, then the successive weights, $w_1, w_2, w_3 \dots \dots$, of the solute extracted will be—

$$w_1 = v_1 s_1 (W - w_1) / n \cdot v_2 s_2,$$

$$w_2 = v_1 s_1 (W - w_1 - w_2) / n \cdot v_2 s_2,$$

$$\dots \dots \dots$$

$$w_n = v_1 s_1 (W - w_1 - w_2 - \dots - w_n) / n \cdot v_2 s_2;$$

and the total weight extracted in the n operations—

$$= w_1 + w_2 + w_3 + \dots + w_n =$$

$$= \frac{v_1 s_1}{n \cdot v_2 s_2} (nW - nw_1 - [n-1]w_2 - [n-2]w_3 - [n-3]w_4 - \dots - w_n) =$$

$$= \frac{v_1 s_1}{v_2 s_2} W - \frac{v_1 s_1}{v_2 s_2} (w_1 + w_2 + w_3 + \dots + w_n) + \frac{v_1 s_1}{n \cdot v_2 s_2} (w_2 + 2w_3 + 3w_4 + \dots).$$

Hence—

$$(w_1 + w_2 + w_3 + \dots) + \frac{v_1 s_1}{v_2 s_2} (w_1 + w_2 + w_3 + \dots) =$$

$$= \frac{v_1 s_1}{n \cdot v_2 s_2} (w_2 + 2w_3 + 3w_4 + \dots) + \frac{v_1 s_1}{v_2 s_2} W.$$

Therefore—

$$w_1 + w_2 + w_3 + \dots = \text{total weight extracted} =$$

$$= \frac{v_1 s_1}{v_2 s_2} W / \left(1 + \frac{v_1 s_1}{v_2 s_2} \right) +$$

$$+ \frac{v_1 s_1}{n \cdot v_2 s_2} (w_2 + 2w_3 + 3w_4 + \dots) / \left(1 + \frac{v_1 s_1}{v_2 s_2} \right) \dots \dots 2.$$

Equation 2, it will be observed, is greater than Equation 1 by the quantity $\frac{v_1 s_1}{n \cdot v_2 s_2} (w_2 + 2w_3 + 3w_4 + \dots) / \left(1 + \frac{v_1 s_1}{v_2 s_2} \right)$, which proves the above statement.

East London Technical College,
London, E.

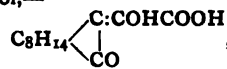
CONDENSATION COMPOUNDS OF CAMPHOR- OXALIC ACID AND AMINES.*

SEVENTH COMMUNICATION ON CAMPHOROXALIC DERIVATIVES.†

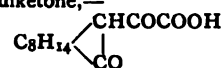
By J. BISHOP TINGLE, Ph.D., F.C.S.,
and
WILLIAM EDWIN HOFFMAN, Jun., Ph.D.

THEORETICAL.

THE investigations carried out by J. Bishop Tingle and his co-workers have shown almost conclusively that the condensation compound of camphor and ethyl oxalate, which has been termed camphoroxalic acid, is an unsaturated keto-alcohol,—

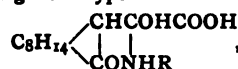


and not the 1,3-diketone,—

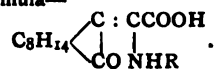


In the course of the work, a new class of compounds was discovered, formed by the condensation of the acid with amines (*Journ. Am. Chem. Soc.*, 1901, xxiii., 363).

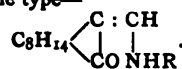
The condensation compounds of the sodium and potassium salts of camphoroxalic acid and of the ethyl ester with amines, both aliphatic and aromatic, were studied. The primary products in all cases were apparently additive substances of the general type—



but only one compound with this formula could be isolated. It was obtained from hydroxylamine and sodium camphoroxalate; the other substances appeared to be unstable and eliminated the elements of water, giving rise to products of the general formula—



Compounds of this nature were prepared from ammonia, hydroxylamine, semicarbazine, aniline, and α - and β -naphthylamine. Such substances as those formulated above might be expected to undergo further change; carbonic anhydride might be evolved with the formation of a compound of the type—



In addition to the above, a third compound of the amine with camphoroxalic acid might be formed, namely, a salt of the acid mentioned above; this would be represented by the formula—



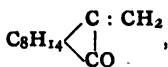
Representatives of these various types were actually prepared from aniline and sodium camphoroxalate. The first by the action of the constituents in a neutral solvent, at the ordinary temperature; the second by the action of aniline and camphoroxalic acid, under pressure at 130°, or by heating the first compound above its melting-point. The third derivative was obtained by the action of aniline on free camphoroxalic acid in a neutral solvent at the ordinary temperature.

In naming these compounds the simplest and most

* From the *American Chemical Journal*, xxxiv., No. 3.

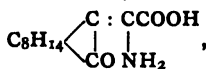
† The previous papers on this subject are as follows:—Inang. Diss., Munich, 1889, p. 34; *Journ. Chem. Soc.*, 1890, lvii., 652; *Am. Chem. Journ.*, 1897, xix., 393; 1898, xx., 318; 1899, xxi., 238; 1900, xxiii., 214; *Journ. Am. Chem. Soc.*, 1901, xxiii., 363.

advisable method appeared to be to regard them as derived from the complex—

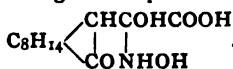


for which the term camphorformene was suggested; it is self-explanatory, and indicates the presence of the double linkage.

By the action of ammonia on sodium or potassium camphoroxalate, sodium or potassium camphorformene-aminocarboxylate was obtained, from which the free acid—

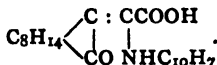


was isolated. Sodium camphoroxalate combines with hydroxylamine, forming the compound—



Semicarbazine unites with potassium or sodium camphoroxalate under the same conditions as hydroxylamine. The products were thought to consist of two compounds having apparently the same empirical formula, but differing in their behaviour towards solvents.

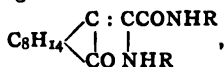
α -Naphthylamine reacted with sodium camphoroxalate under somewhat similar conditions to aniline, and formed α -naphthylcamphorformeneaminocarboxylic acid. The corresponding derivative of β -naphthylamine was also obtained:—



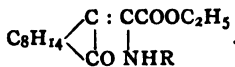
The interaction of orthophenylenediamine and sodium camphoroxalate produced camphorquinoxaline, $\text{C}_{18}\text{H}_{20}\text{N}_2\text{O}_2$.

ETHYL CAMPHOROXALATE DERIVATIVES.

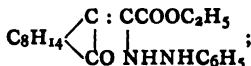
Methylamine, ethylamine, semicarbazine, aniline, and ammonia condensed with ethylcamphoroxalate to form compounds of the general formula—



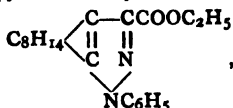
while with aniline or β -naphthylamine the product has the formula—



Phenylhydrazine reacted with ethyl camphoroxalate, forming—



when this was heated above its melting-point, ethyl camphylphenylpyrazolcarboxylate,—

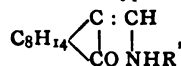


results, and on hydrolysing this compound the free acid was obtained.

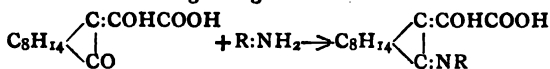
Condensation compounds could not be isolated from ethyl camphoroxalate or sodium camphoroxalate with para- and meta-phenylenediamine, ethylaniline, and dimethylaniline.

The object of the present investigation was to determine, if possible, what action would take place between the free camphoroxalic acid and aliphatic and aromatic amines; to ascertain, as far as we were able, the limits within which the condensation takes place; to endeavour to find the cause of the inhibition of the reaction with some

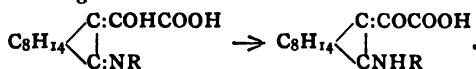
amines, whether it was due to stereometrical influences, to the aliphatic or aromatic nature of the amine, or to its more or less strongly developed positive properties; and to try to discover the conditioning factors for the production of substances of the varying types referred to in the preceding pages. The action of camphoroxalic acid upon a number of metallic compounds was also investigated, and the typical salts of camphoroxalic acid described below have been prepared and analysed. Finally, we desired to investigate the action of acylhalides or substituted acylhalides upon compounds of the type—



the object being to endeavour to obtain evidence of the existence in these compounds of the group R_2NH . Although the behaviour of hydroxylamine and phenylhydrazine towards ethyl camphoroxalate, which has already been referred to, makes it highly probable that the amine attacks the group $>\text{COH}$, yet the possibility of the carbonyl group of the camphor nucleus first reacting with the amine must also be recognised; this, however, would involve the following changes:—



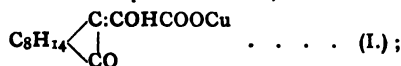
—that is, the :COH group would remain intact, and should be capable of recognition as such, but this was not found to be the case. The alternative suggestion is that intramolecular rearrangement might take place, and result in the change—



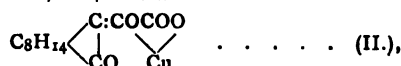
At present there is no evidence in support of this latter view, while the fact that, apart from the salt-forming action of the carboxyl group, camphoroxalic acid combines with amines in equimolecular proportion, would appear to be decisively against it.

METALLIC SALTS OF CAMPHOROXALIC ACID.

When copper nitrate is allowed to react with camphoroxalic acid in a solution of 50 per cent alcohol, at ordinary temperatures, a greenish crystalline mass is obtained, which melts and decomposes at 275° , and has the formula $\text{C}_{12}\text{H}_{14}\text{O}_4\text{Cu}$. The product is the same, irrespective of the proportions in which the copper nitrate and acid are mixed. Two views are possible of the constitution of this salt: if the copper is present in the cuprous condition, it would be—



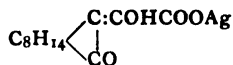
if in the cupric state, the formula—



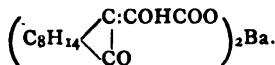
would apply, and analysis could, of course, not distinguish between them. At first sight, Formula I., where the carboxyl group only is affected, would seem to be the more probable, and this view might be thought to derive support from the observation that, when the salt is heated in alcoholic solution at 100° , cuprous oxide is deposited. The view that the salt is represented by Formula II. is based upon the following facts:—Its green colour is characteristic of the cupric but not of the cuprous state. In solution the salt fails to give any indication of the presence of copper ions—which behaviour is entirely in accordance with Formula II. Moreover, unlike the barium salt described below, this one gives no colouration with ferric chloride and alcohol, indicating that a change has taken place in the :COH group of the camphoroxalic acid. The decomposition on heating at 100° is doubtless

analogous to that of Fehling's solution, in which the copper is certainly in the cupric state. According to this view, the compound is a representative of a combination of two distinct types of organo-metallic derivatives, viz., the salt of a carboxylic acid and the metallic derivative of a diketone, such as the sodium derivative of ethyl acetoacetate.

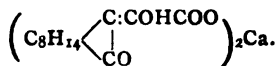
When a solution of silver nitrate in 50 per cent alcohol is allowed to react with camphoroxalic acid, a colourless silver salt,—



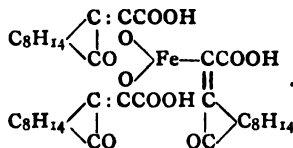
is formed. Under similar conditions, barium nitrate and camphoroxalic acid form a barium salt,—



Calcium nitrate reacts in the same manner, forming the calcium salt,—



In the case of ferric chloride and camphoroxalic acid, the compound which is formed has the characteristic dark reddish violet colour which results in general when ferric chloride acts in alcoholic solution on a compound containing the enol grouping. This substance is somewhat similar to the compounds prepared by R. Schieff, in his work on acetoacetic ester when in combination with benzyl aniline (*Ber.*, xxxi., 205, 601). Its constitution seems to be represented by the formula—



This is based on the results obtained by analysis, and on the fact that the salt dissolves slowly in boiling sodium carbonate solution. It melts at about 85°.

(To be continued).

An Improved Form of X-ray Tube.—Messrs. Heinz, Bauer, and Co., of Berlin, have recently introduced some improvements in X-ray generators, and have sent two of their latest types for experiment; one especially designed for producing rays of great penetration, and the other giving "soft" rays for therapeutic use. In both tubes the anode and cathode are of aluminium, and are fixed opposite each other, the platinum "target" being either quite independent, or—in the case of the penetrating tube—connected to the anode through a bobbin of self-induction. To keep the target cool a new device is adopted, which consists in mounting the platinum disc upon a massive cylinder of copper, and supporting it (like a thimble) upon a thin glass tube, the inner surface of which is open to the air; by these means excessive heating is avoided, even when heavy currents are employed. The bulbs of these tubes are over eight inches in diameter, and the electrical connections are very strongly made. There is in each case an automatic method of regulating, by the liberation of some occluded gas, when the resistance of the tube exceeds a certain limit. The tubes work very well, and produce good sharp shadows, and from their sound construction we consider they will bear a deal of heavy usage.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, January 18th, 1906.

Prof. R. MELDOLA, F.R.S., President, in the Chair.

Concluded from p. 67).

14. "Contributions to the Chemistry of the Rare Earths." (Part I.). By MARIO ESPOSITO.

The methods advocated for the separation of cerium, lanthanum, and "old didymium" have been examined comparatively with the following results.

Preparation of the Material.—Rather more than half a kilogram of finely-powdered Swedish cerite was mixed into a paste with water, and 375 grms. of concentrated sulphuric acid were gradually added. Considerable evolution of heat took place, and the mass swelled up somewhat. The excess of acid was then removed by heat, and the cold white or grey mass was thrown in small portions into 5 litres of ice-cold water. The silica was removed by filtration, and a current of hydrogen sulphide was passed through the solution to precipitate any arsenic, molybdenum, bismuth, copper, or lead which might be present. To the filtrate, oxalic acid was added, which precipitated all the ceria, lanthana, and "didymia" along with lime and traces of the yttrium group which are present in cerite (Brauer, *Trans.*, 1883, xliii., 278). The oxalates were thoroughly washed and dried.

Methods of Separation.—1. The first method tried was that originally proposed by Watts (*Quart. Journ. Chem. Soc.*, 1850, ii., 131). Some of the reddish brown oxide prepared by igniting the foregoing oxalates was boiled for several hours with a strong ammonium chloride solution until no more dissolved. The reddish brown insoluble ceria was washed with ammonium chloride solution, and dissolved by boiling with alcohol and strong hydrochloric acid. The solution showed strong "didymium" absorption bands, and probably also contained lanthanum.

2. Brauer's basic nitrate method (*Trans.*, 1883, xliii., 278; 1885, xlvii., 879) gave fairly good results, but some of the cerium remains in solution.

3. Popp's method (*Annalen*, 1864, cxxxi., 360), which consists either in boiling a neutral solution of the chlorides with sodium acetate and sodium hypochlorite or in passing a current of chlorine through a solution mixed with sodium acetate, gives good results on a small scale, but is inconvenient with larger quantities.

4. Although the same objection applies to Mosander's method with caustic potash and chlorine (*Poggendorff's Ann.*, 1843, lx., 297), very pure ceria can thus be obtained.

5. For ordinary purposes, however, the process devised by H. Debray (*Comptes Rendus*, 1883, xcvi., 928), slightly modified as follows, seems to give rapid and satisfactory results. The dried oxalates were boiled with strong nitric acid until completely decomposed, the solution was evaporated to dryness, the last part of the process being conducted on a sand-bath, and the anhydrous nitrates mixed with twice their weight of sodium nitrate were fused in a porcelain dish until no more nitrous fumes were evolved. The cold mass was detached from the dish, coarsely powdered, and digested in warm water acidified with nitric acid. The insoluble pale yellow ceric oxide was collected and washed with ammonium nitrate solution, as recommended by Schützenberger (*Comptes Rendus*, 1895, cxx., 663). If pure water is used, the finely-divided ceric oxide will pass through the filter. The filtrate was again evaporated to dryness, and fused so as to eliminate the remainder of the cerium. The lanthanum and "didymium" were subsequently separated by fractional crystallisation of the oxalates from strong nitric acid solution, as will be described in a future communication.

6. Very pure ceria can be obtained by the following method, which depends on the use of chromic acid. The

solution of nitrates was precipitated with caustic soda, and the hydroxides were washed several times by decantation with boiling water. A warm aqueous solution of chromic acid was then added, and the whole left for several days. A heavy, yellowish white precipitate remained, which, on ignition at a gentle heat, yielded cerium sesquioxide as an almost white powder without the slightest tinge of red or yellow. When thus obtained, the oxide dissolved readily in boiling hydrochloric acid, and the solution, even when viewed through a considerable thickness, did not show a trace of absorption bands.

This last method will be further studied. A chromic-acid method was formerly devised by Pattinson and Clarke (CHEMICAL NEWS, 1867, xvi., 259), but the process is entirely different from the foregoing, and has the disadvantage of giving a ceric oxide which is absolutely insoluble in acids.

15. "A Synthesis of Aldehydes by Grignard's Reaction." By GORDON WICKHAM MONIER-WILLIAMS.

Gattermann and Maffezzoli (*Ber.*, 1903, xxxvi., 4152) showed that aldehydes could be obtained by the action of organo-magnesium compounds on ethyl formate, $\text{H}\cdot\text{CO}\cdot\text{O}\cdot\text{C}_2\text{H}_5 + \text{IMgX} = \text{C}_2\text{H}_5\cdot\text{O}\cdot\text{MgI} + \text{X}\cdot\text{CHO}$, and various other investigators have carried out analogous syntheses with derivatives of ethyl formate; for example, bromoform, iodoform, ethyl orthoformate, disubstituted formamides, and isonitriles. The author has shown that ethoxymethyleneaniline, $\text{C}_6\text{H}_5\cdot\text{N}:\text{CH}\cdot\text{O}\cdot\text{C}_2\text{H}_5$, reacts readily with organo-magnesium compounds to give the anhydro-compound of aniline with the aldehyde in question, from which the latter is easily obtained. The yields are in many cases far better than those given by the older methods. Ethoxymethyleneaniline was prepared by the action of ethyl iodide on the silver derivative of formanilide, $\text{C}_6\text{H}_5\cdot\text{N}:\text{CH}\cdot\text{OAg}$ (Comstock and Kleeberg, *Am. Chem. Journ.*, 1890, xii., 497), and was allowed to react in absolute ethereal solution at 35° on organo-magnesium compounds. Benzaldehyde, *o*-tolualdehyde, α - and β -naphthaldehydes were obtained from the respective bromo-compounds with yields of 46, 54, 48, and 36 per cent respectively. Some derivatives of β -naphthaldehyde were prepared and analysed, notably β -naphthylacrylic acid, $\text{C}_{10}\text{H}_7\cdot\text{CH}:\text{CH}\cdot\text{CO}_2\text{H}$, and dinitro- β -naphthaldehyde, $\text{C}_{10}\text{H}_5(\text{NO}_2)_2\cdot\text{CHO}$. The method was then applied to the synthesis of a new aldehyde, p -thiophenetylaldehyde, $\text{C}_6\text{H}_5\cdot\text{S}\cdot\text{C}_6\text{H}_4\cdot\text{CHO}$. p -Nitrothiophenetole (Blanksma, *Rec. Trav. Chim.*, 1901, xx., 403) on reduction gave p -thiophenetidine, $\text{C}_2\text{H}_5\cdot\text{S}\cdot\text{C}_6\text{H}_4\cdot\text{NH}_2$, a slightly yellow liquid boiling at 280° . Its acetyl derivative is thiophenacetone, which, however, does not exert the physiological action of phenacetone. The base was successively converted into the iodo-compound, $\text{C}_2\text{H}_5\cdot\text{S}\cdot\text{C}_6\text{H}_4\cdot\text{I}$, a slightly yellow oil boiling at $146^\circ/11$ m.m., and the aldehyde, $\text{C}_2\text{H}_5\cdot\text{S}\cdot\text{C}_6\text{H}_4\cdot\text{CHO}$, boiling at 245° . Several derivatives of the latter were prepared, of which the azine is very characteristic. The oxidation of the aldehyde with alkaline permanganate gave rise to a sulphonecarboxylic acid, $\text{C}_2\text{H}_5\cdot\text{SO}_2\cdot\text{C}_6\text{H}_5\cdot\text{CO}_2\text{H}$.

16. "The Action of Ultra-violet Light on Moist and Dry Carbon Dioxide." By SAMUEL CHADWICK, JOHN EDWIN RAMSBOTTOM, and DAVID LEONARD CHAPMAN.

By the action of ultra-violet light on dry carbon dioxide, the authors have succeeded in partially decomposing carbon dioxide into carbon monoxide and oxygen. The circumstances of the experiment rendered it impossible to decide what percentage of the oxygen was present in the form of ozone. If the carbon dioxide is moist there is no decomposition. The peculiar influence of water vapour is believed to be due to the action which it exerts on the vibrations of the electrons of atoms of which the molecules of carbon monoxide, carbon dioxide, and oxygen are composed. The facts, discovered by Chapman and Burgess, concerning the induction period of a mixture of chlorine and hydrogen are regarded as confirming this view.

17. "A Contribution to the Study of Stable Diazo-compounds." (Preliminary Note). By GILBERT THOMAS MORGAN and WILLIAM ORD WOOTTON.

p -Aminobenzaniline, which has been shown to give on diazotisation a stable diazo-carbonate and a corresponding nitrite (*Trans.*, 1905, lxxxvii., 938), also furnishes a diazonium picrate, $\text{C}_6\text{H}_5\cdot\text{CO}\cdot\text{NH}\cdot\text{C}_6\text{H}_4\cdot\text{N}_2\cdot\text{O}\cdot\text{C}_6\text{H}_2(\text{NO}_2)_3$, which is precipitated even in the presence of hydrochloric acid. This substance, which is insoluble in water and crystallises from warm alcohol in rosettes of yellow needles decomposing at 132° , does not explode on percussion. When dissolved in aqueous caustic soda and added to alkaline β -naphthol, it yields a precipitate of benzoyl- p -aminobenzeneazo- β -naphthol. Mercury acetamide has been found to yield sparingly soluble, stable additive compounds when added to the aqueous solution of a diazonium salt, and a series of these substances is under investigation. The compound with benzenediazonium chloride gave, on analysis, $\text{N} = 12.46$, whereas $\text{C}_6\text{H}_5\cdot\text{N}_2\text{Cl}\cdot\text{Hg}(\text{NH}\cdot\text{CO}\cdot\text{CH}_3)_2$ requires $\text{N} = 12.26$ per cent. Similar stable mercury acetamide derivatives have been obtained from the substituted anilines and their homologues.

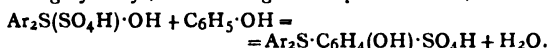
18. "Triarylsulphonium Bases." By SAMUEL SMILES and ROBERT LE ROSSIGNOL.

Quite recently Kehrman and Duttenhöfer have published a paper (*Ber.*, 1905, xxxviii., 4197) dealing with mono- and di-arylsulphonium bases, and, although their work does not clash with that of these investigators, the authors considered it desirable to communicate the following preliminary note to the Society.

It has been found that triarylsulphonium bases can be prepared in three ways.

1. By the action of thionyl chloride on a phenol or phenolic ether in presence of aluminium chloride.
2. By the action of a sulphinic acid on a phenolic ether in presence of concentrated sulphuric acid.
3. By the condensation of an aromatic sulfoxide with phenols or their ethers.

It can be shown that sulfoxides are formed during the first and second reactions, and hence the third reaction may be regarded as the final stage of the first two. The condensation between sulfoxide and phenol takes place with acid condensing agents, and it is the authors' view that the sulfoxide, being basic in character, first forms a salt $\text{Ar}_2\text{S}(\text{SO}_4\text{H})\cdot\text{OH}$, which then reacts with the phenol, losing hydroxyl, and forming the sulphonium salt, thus:—



This reaction seems to be reversible, and a slight excess of phenol must be present to render the conversion of sulphoxide to sulphonium salt complete. At present only the hydroxy- and ethoxy-derivatives have been examined, but the authors hope to extend the investigation to amide and other compounds.

19. "An Improved Apparatus for the continuous Extraction of Liquids with Ether." By RICHARD SISSON BOWMAN.

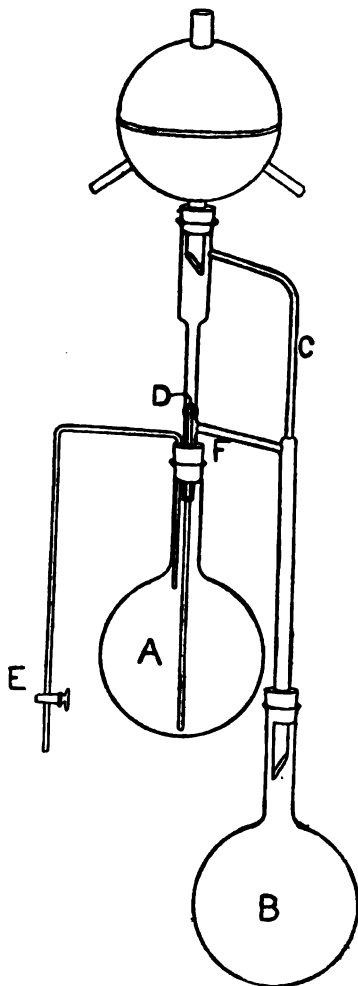
The apparatus here described differs essentially from those due to Hagemann (*Ber.*, 1893, xxvi., 1975), Pellizza (*Chem. Centr.*, 1904, i., 851), and Mameli (*Chem. Centr.*, 1905, ii., 106). In the latter, hot ether vapour is blown through the liquid to be extracted, whilst in the former the extraction is effected by passing cool liquid ether through the solution. Violent bubbling is thus avoided, together with the consequent risk of carrying over mechanically into the ether flask some of the liquid to be extracted.

The apparatus comprises a simple system of tubes, a condenser, and two ordinary flasks, preferably round bottomed.

The liquid to be extracted is placed in the flask A in sufficient quantity to very nearly fill the flask to the bottom of the neck. This flask may frequently be replaced with advantage by a wide test-tube. From one-eighth to one-fifth of this volume of ether, with which the substance is to

be extracted, is placed in the flask B, which is heated on a water-bath or electric heater. The ether passing as vapour up tube C is condensed in the condenser, and falls back and collects in the inner tube, D. A column presently accumulates in D long enough to force its way through the liquid in A, and there is then a constant succession of small drops of ether ascending in A, collecting on the surface of the liquid, and finally overflowing at F and returning to the flask B.

On completion of the process of extraction, the neck of the flask will be filled with ether. There will also be a column of ether in tube D, reaching a few centimetres above tube F, and hence the neck of the flask may be emptied by opening the stopcock E and allowing the ether to run off into some vessel. If necessary, the flask may now be replaced by a similar one containing a fresh portion of the liquid to be extracted, and the process resumed.



Among other advantages claimed for this apparatus are that, whereas the other forms involve either expert glass-blowing or innumerable cork and indiarubber joints, it is simple in construction, and may easily be made by anyone moderately skilled in the use of the blowpipe. It is very easy to keep clean and convenient to charge, since the vessels which contain the liquid to be extracted and the extracting liquid are ordinary flasks. A large quantity of liquid, moreover, may be rapidly and completely extracted with the use of a very small quantity of ether.

Benzene, ligroin, or any other suitable solvent may be used with equal ease, provided it is immiscible with water and of lower specific gravity than the liquid which it is to be used to extract.

Ordinary Meeting, February 1st, 1906.

Prof. R. MELDOLA, F.R.S., President, in the Chair.

MR. J. B. TILLOTT was formally admitted a Fellow of the Society.

Certificates were read for the first time in favour of Messrs. Roderick Harold Capper Birt, B.A., 54, Shooter's Hill Road, Blackheath, S.E.; William Dickson, Branksome, Bridge of Weir; James Stuart Hills, Oxford Street, W.; Ernest Ormerod, M.Sc., Ph.D., 8, Leopold Road, Wimbledon, S.W.; John Henry West, 11, Sydney Street, Chelsea, S.W.; Henry John Wiffen, 17, Albany Road, Manor Park, E.

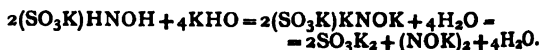
Of the following papers, those marked * were read:—

*20. "Hydroxylamine- $\alpha\beta$ -disulphonates (Structural Isomerides of Hydroximinodisulphates or Hydroxylamine- $\beta\beta$ -disulphonates)". By TAMEMASA HAGA.

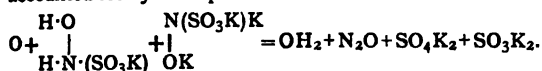
In a previous paper (*Trans.*, 1904, lxxxv., 78; *Proc.*, 1903, xix., 281), it was shown that Fremy's metasulphazilates are hydroxylaminetrisulphonates. From these salts there has now been obtained by hydrolysis a new series of salts which are structurally isomeric with Fremy's neutral and basic sulphasotates, now known to be hydroxylaminedisulphonates. The new salts are decomposed by sodium amalgam, and are proved by the nature of this change, by their decomposition by concentrated potassium hydroxide solution, and by their hydrolysis, to be hydroxylamine- $\alpha\beta$ -disulphonates, in contrast with the sulphasotates, which are hydroxylamine- $\beta\beta$ -disulphonates $\text{SO}_3\text{K}\cdot\text{ONH}\cdot\text{SO}_3\text{K}$ and $\text{HON}(\text{SO}_3\text{K})_2$, respectively. This is believed to be the first indisputable case of the occurrence of fundamental structural isomerism among inorganic compounds. Sodium converts the new salts into sulphate and aminemonosulphonate; potassium hydroxide into the same products, together with nitrogen; and acids transform them into sulphate and hydroxylamine. The new salts are practically all soluble in water and fairly stable; those prepared are principally the di- and tri-potassium salts, di- and tri-sodium salts, and the di-ammonium salt. A hydroxylamine- α -monosulphonate seems incapable of existence; it would be, for example, a sulphate of potassium and amidogenium, $\text{H}_2\text{NO}\cdot\text{SO}_2\cdot\text{OK}$.

DISCUSSION.

Dr. DIVERS said that it follows somewhat unexpectedly from the author's results that it is the presence of oxylic hydrogen and not aminic hydrogen in a hydroxylamine derivative or in hydroxylamine itself which makes it easily oxidisable. For, whilst a hydroxylamine- $\alpha\beta$ -disulphonate is not sensitive to oxidising agents, although it contains an atom of aminic hydrogen, a hydroxylamine- $\beta\beta$ -disulphonate which contains none is readily oxidised to a peroxylamine-sulphonate (*Trans.*, 1904, lxxxv., 78; *Proc.*, xix., 281) by virtue of the oxylic hydrogen atom, which is present in it but not in the $\alpha\beta$ -salt. Applying this deduction from facts to the case of the very sensitive hydroxylaminemonosulphonates, an explanation is found for the fact that these salts, when oxidised, as by cupric chloride or by cupric oxide in alkaline solution (*Trans.*, 1889, lv., 760), give nitrous oxide but no hyponitrite, although when decomposed by potassium hydroxide alone they give much hyponitrite, together with sulphite. The production of hyponitrite by alkali alone is represented by—



its non-production, when cupric oxide is also present, is accounted for by the equation:—



(Copper sulphate induces oxidation of a hydroxylamine-monosulphonate catalytically, causing one-half of it to be reduced to aminemonosulphonate in oxidising the other half in the way just formulated. Compare *Trans.*, 1900, lxxvii., 978).

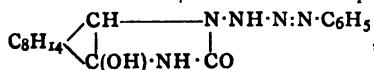
It should therefore be taken into consideration, when determining the constitution of organic and other substitution derivatives of hydroxylamine, that indifference to oxidising agents is not evidence of the absence of aminic but of oxylic hydrogen.

He also suggested, in explanation of the fact that a small part of a hydroxylamine- α -disulphonate is much more difficult to hydrolyse than the rest, that the salt may polymerise to some extent during the heating, and thus become resistant to hydrolysis.

In reference to the circumstance that some of the author's results had been anticipated by Raschig (*Ber.*, 1906, xxxix., 245), and the question of dating communications have been raised by Prof. McLeod, Prof. ARMSTRONG expressed the hope that no alteration in the existing practice would be made, and that nothing would be done to deprive authors of the liberty which they had so long enjoyed of correcting their papers up to the moment of publication. It was most undesirable that anything should be done to encourage discussions as to priority among authors. The public were gainers, when, as in the present case, independent workers arrived at similar results.

*21. "Studies in the Camphane Series. Part XXI. Benzenediazo- ψ -semicarbasinocamphor and its Derivatives." By MARTIN ONSLOW FORSTER.

When a diazonium salt is added to an aqueous solution of camphoryl- ψ -semicarbazide nitrate, the corresponding derivative of benzenediazo- ψ -semicarbasinocamphor,—



is precipitated. Compounds of this class may be called diazo- ψ -semicarbazines, and have been obtained from diazotised aniline, *p*-toluidine, *p*-anisidine, *p*-chloroaniline, *p*-bromoaniline, and the three nitroanilines; they are characterised by the readiness with which dilute alkalis resolve them into camphoryl- ψ -carbamide and the corresponding phenylazoimide. The author dissented from the views on coloured camphor derivatives expressed by Armstrong and Robertson (*Trans.*, 1905, lxxxvii., 1272).

DISCUSSION.

In replying to Dr. Forster, Prof. ARMSTRONG said that, beyond remarking that Stobbe's results were in no way irreconcilable with his (the speaker's) views as to the origin of colour, as he proposed to consider them on another occasion, he should not deal with them then; nor would he continue the discussion on the relation of optical activity to structure in hydrazones, as this subject could not be dealt with properly in the time at disposal. He would confine his remarks to the compounds described by Dr. Forster. Of the two alternative formulæ available for the semicarbazines, Dr. Forster had selected that containing the group $\text{N}\cdot\text{NH}\cdot\text{N}\cdot\text{NPh}$. Inasmuch as compounds containing the group $\text{N}\cdot\text{NPh}$ were at least yellow, he was of opinion that the formula selected did not represent the colourless compounds. He was of opinion also that, although prepared by one method, Dr. Forster's compounds were not all of one type, any more than *o*- and *p*-nitrophenol and nitraniline were compounds of the same type as the corresponding bromophenols and bromanilines. It had long been admitted that the nitro-compounds referred to are at least partially composed of the isodynamic

quinonoid forms; the differences observed by Dr. Forster between his colourless and coloured compounds were precisely those observed on comparing the bromanilines with the nitranilines.

Dr. LANDER remarked that it was unlikely that nitrogen compounds of this type would exhibit tautomerism. Pechmann had shown that the so-called "virtually tautomeric" mixed amidines were mixtures or "solid solutions" of structural isomerides, and further that the isomerism disappeared when the hydrocarbon residues were of different types, as in Dr. Forster's compounds.

Dr. LOWRY said that Dr. Forster appeared to have overlooked the fact that one of Stobbe's compounds, the phenyldimethylfulgide (*Ber.*, 1905, xxxviii., 3895) existed both in a yellow and in a colourless form, the two forms being convertible into one another in much the same way as the isomeric white and yellow oximes of the mesoxamide series studied by Dr. Whiteley (*Trans.*, 1903, lxxxiii., 24). There was, therefore, the clearest possible evidence of the existence of dynamic isomerism in Stobbe's series of compounds, and the argument that individual members of such a series must have the same constitution because prepared in the same way and exhibiting similar chemical properties was entirely fallacious.

Dr. MORGAN cited the case of the ortho-diazoimines as another example of a group of compounds the members of which series differed considerably in colour although they were all prepared by the same general method and had similar properties. Diazoiminobenzene and its acyl derivatives were white or colourless substances. 2:3-Diazoiminonaphthalene was described as yellow, whereas 1:8-diazoiminonaphthalene is brownish red. In spite of this colour difference there seemed to be no reason for supposing that these substances were differently constituted.

Although three of Dr. Forster's diazo- ψ -semicarbazines were nitro-compounds and possibly owed their colour to the presence of the nitro-group, yet it was very unlikely that the yellow *p*-anisyl derivative should be differently constituted to the white *p*-tolyl compound, inasmuch as the former differed from the latter only in the substitution of methoxyl for methyl in the para-position to the diazo-compound.

Dr. FORSTER, in reply, regretted that Dr. Lowry had misunderstood his attitude. He had not used "the argument that individual members of such a series must have the same constitution because prepared in the same way and exhibiting similar chemical properties." His contention was, that in the absence of chemical distinctions among a series of compounds prepared alike, it was unsafe to say that the structural type of a colourless compound must differ from that of a coloured one merely because the substances differed in appearance. That was his reason for criticising the views of Professor Armstrong and Mr. Robertson regarding the structure of their camphor-quinonehydrazones, and nothing had been said that evening which impaired his conviction that to deduce a certain constitutional formula for a coloured substance solely from the assumption that the colour is due to the constitution ascribed is arguing in a circle, and therefore unacceptable.

*22. "The Relations between Absorption Spectra and Chemical Constitution. Part I. The Chemical Reactivity of the Carbonyl Group." By ALFRED WALTER STEWART and EDWARD CHARLES CYRIL BALY.

Attention has been drawn (*Trans.*, 1904, lxxxv., 1029, and 1905, lxxxvii., 766) to the relation which exists between tautomerism and the selective absorption which is shown by the spectra of derivatives of ethyl acetoacetate, whilst it has also been proved that the rates of addition of sodium hydrogen sulphite to ketones can be influenced by the introduction of a carboxyl radicle into the molecule, and that the effect thus produced is directly opposite to what one would expect if the hypothesis of steric hindrance held good (*Trans.*, 1905, lxxxvii., 185, and *Proc.*, 1905, xxi., 78). In the present paper, it is shown that these two phenomena are connected with one another, and the relation is indicated

between the activity of the carbonyl group in certain ketones and their selective absorption. It is pointed out that in certain cases the phenomena of tautomerism furnish an explanation of the exceptional reactivity of the carbonyl group, as the passage from the enolic to the ketonic form would give rise to a carbonyl radicle, the condition of which may be considered as approximating to that of a nascent atom. This "nascent carbonyl group" would be more reactive than the normal carbonyl. But although in most instances tautomerism suffices to explain the phenomena, it breaks down in several cases, and to cover these a new and wider conception is required. From spectroscopic evidence it appears that in the α -diketones a vibration is going on which to a certain extent resembles that which was found in the case of ethyl acetoacetate and its derivatives. The nature of this vibration cannot be easily expressed in the ordinary structural formulæ without the possibility of misconception, but it may be indicated somewhat as follows:—The vibration is brought about by some change in the relations between the carbon and oxygen atoms, and it in some respects resembles the transition from the ketonic to the enolic form and back again. Using this analogy, we may postulate that the two extreme phases of the vibration can be represented by the formulæ:—



The conversion of I. into II. would give rise to the "nascent carbonyl group" just as the change of the enolic into the ketonic form does. Further, the experimental evidence available shows that some such relation does actually exist between the carbonyl groups of benzoquinone, and the great reactivity of the quinones can be thus explained. It is proposed to term the general phenomenon "*isorropesis*," and to call "*isorropic*" those radicles the activity of which is thus produced.

(To be continued).

SOCIETY OF CHEMICAL INDUSTRY. (LONDON SECTION).

Meeting held at Burlington House on February 5th, 1906.

"Carburetted Water-gas in the Bunsen Burner." By MATSUMI CHIKASHIGE.

In a previous paper (*Journ. Soc. Chem. Ind.*, 1904, 50) H. Matsumoto and the author pointed out the defects of water-gas as a fuel in the chemical laboratory. Carburetted water-gas is now prepared in the Kyoto University by injecting heavy petroleum oil with steam into a water-gas generator filled with ignited coke. The gas produced is passed through a superheater loosely packed with fire-bricks, and then through a scrubber, after which treatment it enters the gas-holders. The mean composition of the gas differs little from that of coal-gas, and the products of combustion closely resemble those of coal-gas.

To obtain the usual flame of a Bunsen burner (15 c.m. long) a flow of 120 litres of coal-gas, 300 of water-gas, and 120 of carburetted water-gas are required; therefore the triangular orifice of the ordinary Bunsen burner for coal-gas requires to be only slightly enlarged to get the same sized flame with the carburetted water-gas.

The carburetted gas has no effect on the ordinary laboratory vessel, and the products of combustion, unlike those of plain water-gas, are not more injurious in insufficiently ventilated laboratories than those of coal-gas.

"The Loss of Nitre in the Chamber Process." By J. K. H. INGLIS.

The loss of nitre, which amounts in many works to about 3 per cent of the sulphur burnt, can best be traced by complete analyses of the flue gases in which the quantity

of nitre, calculated as nitric oxide, should amount to about one part in one thousand by volume. The analysis of these gases cannot be carried out by means of aqueous absorbents owing to the formation of complicated bodies by the interaction of nitrous acid and sulphur dioxide. But the whole analysis can be carried out very conveniently by the fractional distillation of the gases, first at the temperature of liquid air, and subsequently at higher temperatures. The results showed that only about 4 per cent of the lost nitre was lost as nitrous oxide, and that as a mean of eight experiments the loss as nitrogen peroxide amounted to about 43 per cent of the total loss. In the first experiments the temperature was not sufficiently low to effect the separation of nitric oxide from the flue gases, since at the temperature of liquid air the vapour pressure of nitric oxide is considerable. Some further experiments were therefore made at a still lower temperature, obtained by making the liquid air boil under diminished pressure. Contrary to expectation, however, the amount of nitrogen oxides found was no greater than that in the earlier experiments; and this might therefore be taken to mean the nitric oxide is not present in the flue gases. These last experiments, however, were not made under quite the same conditions of the plant as the earlier experiments, and further work is needed before one can draw any definite conclusion in regard to the presence of nitric oxide.

"The Removal of Nitrous Acid from Concentrated Nitric and Sulphuric Acid." By O. SILBERRAD and B. J. SMART.

The experiments were carried out at the Royal Arsenal, Woolwich, and communicated with the consent of the Explosives Committee. They were made to determine to what extent the reaction between nitrous acid and amines or amides occurs in concentrated acids. Nitric acid containing a small percentage of nitrous acid was taken either alone or in admixture with sulphuric acid. Various reagents which are known to react readily with nitrous acid in aqueous solution were added to the concentrated acid, and allowed to stand at the ordinary temperature. From time to time the amount of nitrous acid present was determined by titration with permanganate. The addition of hydrazine occasions an explosion, and with this exception substances such as urea, lead peroxide, oxamide, methylamine nitrate, and amido-guanidine, are very inert towards nitrous acid in presence of concentrated nitric acid, although they react readily enough in dilute solution. The observation of Franchimont that urea nitrate decomposes with evolution of carbon dioxide and nitrous oxide was confirmed.

FARADAY SOCIETY.

Ordinary Meeting, January 30th, 1906.

Prof. A. K. HUNTINGTON in the Chair.

THE following nominations for the Officers and Council to be elected at the forthcoming Annual General Meeting were announced:—

President—Lord Kelvin, O.M., G.C.V.O., F.R.S.
Vice-Presidents—Prof. A. Crum-Brown, M.D., F.R.S.; Prof. W. Hittorf; Sir William Huggins, O.M., K.C.B., F.R.S.; Sir Oliver Lodge, F.R.S.; Ludwig Mond, Ph.D.; F.R.S.; Lord Rayleigh, O.M., P.R.S.; Prof. J. J. Thomson, F.R.S.

Treasurer—F. Mollwo Perkin, Ph.D.
Council—Bertram Blount, F.I.C.; W. R. Cooper, M.A.; Prof. A. K. Huntington; R. S. Hutton, D.Sc.; Herbert Jackson, M.A.; T. M. Lowry, D.Sc.; W. M. Morrison, M.Inst.C.E.; O. J. Steinhart, Ph.D.; J. Swinburne, M.Inst.C.E.; Prof. E. Wilson.

Mr. W. MURRAY MORRISON read an abstract of a paper presented by ADOLPHE MINET on "The Electric Furnace:

its Origin, Transformations, and Applications." (Part III.).

The previous sections of the paper, which is of a classificatory and historical character, dealt with the first or laboratory period of the development of the electric furnace. The second period, inaugurated by the researches of the brothers Cowles, and of Héroult (1886-1887), saw the birth of industrial furnaces, and the beginnings of three new industries—the electrometallurgy of aluminium, the electrometallurgy of sodium, and the preparation of refractory materials. The present paper deals almost entirely with the first of these industries.

The methods for the manufacture of aluminium are divided into two principal groups—the electrothermic methods, which produce the metal alloyed with copper or iron, or combined with carbon, and electrolytic methods, which yield pure aluminium. The furnaces of Cowles, Borchers, and Willson, the early types of the Héroult furnace, and the Minet "mixed" furnace are described, among others, under the former heading. The principal electrolytic furnaces dealt with are those of Héroult, Minet, and Hall, and finally some of the more recent suggested improvements in the electrometallurgy of aluminium are briefly outlined.

Mr. E. RISTORI added some interesting details in connection with the early work of Héroult and others. Kleiner, in 1885, had unknowingly decomposed bauxite, and produced small quantities of aluminium.

Mr. W. M. MORRISON pointed out that Héroult's invention was actually made in 1885, although his patents only dated from 1887.

Dr. O. J. STEINHART referred to recent patents of Hall for purifying bauxite electrolytically, and of Bett for depositing pure aluminium from an impure electrolyte.

Mr. BERTRAM BLOUNT said that, although the purification of the bauxite was the whole crux of the aluminium problem, yet now there might be found uses for impure aluminium in connection with "thermit" welding. He discussed some of the developments that had taken place in the general design of electric furnaces.

Mr. F. W. HARBORD, in reply to a question, said that calcium might, if it could be made cheaper, replace aluminium for getting rid of over-oxidation in steel, but it had not yet been really tried.

Dr. J. A. HARKER then gave a demonstration of a Solid Electrolyte Tube Furnace.

The furnace was designed for uses in which the presence of hydrocarbons or other reducing gases, inseparable from a carbon tube furnace, was undesirable. It has, in particular, been used for calibrating thermo-couples, and determining the melting-point of platinum, which the author gives as 1703—1713° C. The furnace is practically an overgrown Nernst lamp, consisting of a tube made of a mixture of zirconia and 10 per cent yttria, which is squirted through a die and then baked. The small type shown in operation was 2½ in. long between the contacts. When taking one ampere at about 120 volts a temperature of 1600° C. was attained. The tube is made conducting either by heating direct, or else by surrounding it with an external nickel heating coil, separated from it by a layer of pure zirconia, the combination forming a "cascade" type of furnace. Special attention has to be paid to the contact of the platinum leading-in wires and the tube.

With regard to the mechanism of conduction through Nernst conductors, the author thought that a comprehensive and satisfactory theory was at present lacking. It was, however, certain that the conduction was electrolytic, because by pumping away the atmosphere surrounding a Nernst glower, to prevent re-combination, a metallic filament could ultimately be obtained.

Mr. BERTRAM BLOUNT thought no special mechanism was needed to explain solid electrolysis. Ordinary transference of ions would take place in a semi-plastic mass.

Dr. T. M. LOWRY referred the author to the dissertation by Reynolds. The conduction was similar to that which occurred in hot glass.

Mr. E. B. R. PRIDEAUX communicated a paper entitled "Note on the Production of Ozone by Electrolysis of Alkali Fluorides." (See CHEMICAL NEWS, xciii., p. 47).

NOTICES OF BOOKS.

The Nature and Origin of Living Matter. By H. CHARLTON BASTIAN, M.A., M.D., F.R.S., F.L.S. London: T. Fisher Unwin. 1905.

THIS volume contains a full account of Dr. Bastian's views, which are already well known in outline by the scientific world. The author has now brought together a large accumulation of material, which he has been collecting for many years, and he gives minute descriptions of his investigations and observations, fully illustrated by photo-micrographs. A large part of the book is devoted to the consideration of the subject of archebiosis, and by the experiments described the author believes that he has proved that no logical or consistent reason exists for a disbelief in its present occurrence. Then he passes to Heterogenesis and the Heterogenetic Origin of Bacteria and their Allies, and gives details of many miscellaneous examples of heterogenesis. While every page of the book reveals the writer's talents for investigation, and the excellence of his equipment for work of this nature; nevertheless, it is doubtful whether it will lead to the conversion of many, although through it some may perhaps be brought to recognise at any rate the possibility of the truth of what Dr. Bastian puts forward. Difficult as the facts may be of credence, an open mind must be preserved towards them; but it may be pointed out that a certain weakness is displayed in discussing the interpretation of some of the phenomena observed; for instance, the critical flask experiments. A weak point seems to be the "necessary precautions against infection," and the impossibility of maintaining absolute isolation of the organisms under observation, which, of course, leaves a loophole for explaining away the phenomena. Every one interested in the problem of the origin of life should devote serious and careful study to this book; all new theories must meet with opposition, but the vigour of the opposition is often a measure, not of the falsity of the new hypothesis, but of the depths of our ignorance of some aspect of the subject before it was propounded.

Chemie der Alicyclischen Verbindungen. ("The Chemistry of the Alicyclic Compounds"). By OSSIAN ASCHAN. Braunschweig: Friedrich Vieweg und Sohn. 1905.

THE name suggested by Bamberger for those organic substances which contain a ring nucleus and exhibit the essential characteristics of aliphatic compounds, has been adopted by the author as the most suitable and convenient for the cyclo-paraffins of general formula C_nH_{2n} and their derivatives, excluding naturally the aromatic hydrocarbons; and this is the first book yet published devoted entirely to the study of these substances. The author discusses in it the influence of the ring binding upon both the chemical and physical nature of the compounds and their stereo-chemistry, besides describing in the special part their methods of formation. The alicyclic compounds form a class which is of great importance in organic chemistry, many of the substances being employed technically, and special attention is devoted to such as are of more than purely theoretical interest. These include the camphors, terpenes present in ethereal oils and the naphthenes of petroleum, besides many other naturally-occurring compounds. The author, who has done pioneering work upon the alicyclic series, has also rendered a valuable service to organic chemistry in collecting and arranging systematically all the results which have been obtained in this region.

An Introduction to Volumetric Analysis. By A. JAMIESON WALKER, Ph.D. (Heidelberg), B.A., and OWEN E. MOTT, Ph.D. (Heidelberg). London: Chapman and Hall, Ltd. 1905.

WE have in this little book a useful course of volumetric analysis, presented in such a way as to force the student to think about what he is doing, and to prevent him working mechanically. With this end in view, the authors have as far as possible not given exact directions as to weights and volumes to be used, but simply provided the data necessary for the student to calculate these quantities, and one useful feature is the system of giving many tables of strengths of solutions, left blank to be filled up by the worker. The directions for the experiments seem to be in all cases clear and full, equations are given wherever necessary, and the calculations are explained very carefully. The order in which the subject is attacked hardly seems to be the best possible; it is generally found advisable not to give the student solutions of known strength to work with, but to let him first prepare standard solutions, and, working with them, to determine unknown solutions. This, however, is a criticism which only applies to the first few pages, and it would not be difficult to alter the arrangement of the experiments if it seemed desirable.

Kurses Lehrbuch der Organischen Chemie. ("A Short Text-book of Organic Chemistry"). By Prof. Dr. A. BERNTHSEN. Ninth Edition. Braunschweig: Friedrich Vieweg und Sohn. 1906.

THE English version of Prof. Bernthsen's "Text-book of Organic Chemistry" has been much used in schools and colleges in this country, and a ninth edition will be heartily welcomed both by students and teachers. Like the eighth, this edition has been prepared in collaboration with Dr. Ernst Mohr, and some important changes have been made in it. For instance, the text is for the first time divided into three parts, a procedure rendered necessary by the growth of our knowledge of compounds belonging to the heterocyclic class formerly treated partly in the chapter on the transition to the aromatic compounds, and partly in the appendix to the benzene derivatives. These heterocyclic compounds now form a large and important class to themselves, while the cyclic polymethylenes are classified together with the benzenes and hexamethylenes as isocyclic substances. With these exceptions the work preserves very much its original form and distinctive character, the excellent summaries and generalisations, which were so greatly to be commended, being still a prominent feature of the book.

The Conductivity of Liquids. By OLIN FREEMAN TOWER, Ph.D. Easton. Penna.: Chemical Publishing Company. 1905.

ONE of the aims of this book, which describes methods, results, chemical applications, and theoretical considerations, is to lead to the more general adoption of the Kohlrausch system of units, and in it the author gives a very clear and concise summary of the work which has been done on the conductivity of liquids. All standard methods are well described, and special attention is paid to transport numbers and the theory of electrolytic dissociation. One valuable portion of the book is devoted to non-aqueous solutions and the conductivity of electrolytes in mixed solvents, both subjects being very lucidly treated.

Académie des Sciences.—At the meeting on the 12th of February the Academy of Sciences elected Sir William Crookes a Correspondent of the Institut (*Académie des Sciences*, General Physics Section), in place of M. Bichat, deceased.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances de l'Académie des Sciences. Vol. cxlii., No. 3, January 15, 1906.

The Component $\dot{C}(OH)$ of Tertiary Alcohols.—Louis Henry.—The alcohol function resides in the component $\dot{C}(OH)$ of tertiary alcohols, especially in trimethylcarbinol, $(H_3C)_3C(OH)$, the perfect type of this class of compounds. The author makes a series of experiments showing how the presence of elements or different radicals attached to the carbon of the component $\dot{C}(OH)$ profoundly modify the functional nature of the hydroxyl— $\dot{O}H$ alcohol.

Copper Silicide and a New Method of Formation of Silicon Soluble in Hydrofluoric Acid.—Paul Lebeau.—In a previous paper the author showed that the limit of combination of copper and silicon in industrial cupro silicon containing 50 to 60 per cent of silicon never passes 10 per cent, and apparently corresponds to the production of a definite compound having a formula $SiCu_4$. He now investigates what is the influence the velocity of cooling has on a molten mixture of copper and silicon. Also the total proportion of silicon at the limit of silicuration, but his results do not admit of the statement of any fixed law. The limit of silicuration, however, does correspond to $SiCu_4$.

Thorium Silicide.—O. Hönigschmid.—It was predicted that thorium oxide would be reduced by silicon at the high temperature of the electric furnace. The author makes several attempts by this method, hoping to obtain thorium silicide which had never been prepared. He finds, however, that even with a large excess of silicon a considerable proportion of thorium remains unattacked. The product obtained looks like melted metallic silicon. When treated with potash the excess of silicon can be removed, and a grey powder remains which, examined under the microscope, looks like crystalline fragments and non-reduced oxide. The metallic portion is attacked by all the mineral acids. It is impossible to separate this from the oxide, which only hot concentrated sulphuric acid can dissolve. The use of aluminium, however, will dissolve the silicon without entering into combination with it, and so allows of the preparation of crystalline thorium silicide. There forms at the same time an alloy of thorium and aluminium of a different crystalline form.

Diamine Diazoics.—Léo Vignon.—The author obtains by linkage with aniline a series of new compounds, and enunciates the following law relative to the diazotation of the aromatic diamines:—The diazotation of the two NH_2 groups of the diamines takes place in the same manner as that of the monamines when the NH_2 groups are joined to distinct benzenic radicals. When the two groups are joined to the same radical the diazotation is not effected in the case of the ortho-compound, whilst in the case of the meta- and para-derivatives very unstable diazoic derivatives are formed.

Estimation of Small Quantities of Chloroform (1) in Air, (2) in Blood, or an Aqueous Liquid.—Maurice Nicloux.—The author describes an application of the classic reaction of M. Dumas,—



to the estimation of small quantities of chloroform, avoiding the complication of a sealed tube. In order to estimate the chloroform in the water vapour of the air, this is collected in alcohol, and to apply the same method to the estimation of the chloroform present in blood or an aqueous solution he describes certain modifications.

Estimation of Carbonic Oxide in Air by means of Iodic Anhydride.—Albert Lévy and A. Pécoul.—The authors find that during the important researches on the subject of the combustible gases in air very small traces of carbonic oxide give an intense colouration with starch when acting upon iodic anhydride, whilst 1 volume in 10,000 of acetylene produces no colouration.

Combustion of Acetylene by means of Oxygen.—Paul Mauriceau-Beaupré.—The author finds that in the analysis of industrial atmospheres there are difficulties in the way of combusting the acetylene present with oxygen, and he prefers to utilise MM. Albert Lévy and A. Pécoul's apparatus.

Direct Proportion between the Cryoscopic-point of a Mineral Water of the Bicarbonate Class and the Composition of this Water expressed in Anhydrous Salts and Mono-carbonates.—Lucien Graue.—The author's researches on the cryoscopic of mineral waters allow him to determine in a perfectly definite manner the relation existing between the cryoscopic-point of a mineral water of the bicarbonate class, such as that of Châtel-Guyon, and its composition. The analyses of mineral waters generally are usually reduced to bicarbonates, and there apparently exists no relation in the ordinary way between the total of their mineral matter and their cryoscopic-points. However, he definitely proves that if in the case of bicarbonate waters their composition is expressed in anhydrous salts and mono-carbonates there is a direct proportion between the cryoscopic-point and the composition.

MISCELLANEOUS.

On Anti-friction Metals.—Sergius Kern, M.E., St. Petersburg.—In the CHEMICAL NEWS, No. 2410 (xciii., p. 47), was inserted my note on anti-friction metals. I use the results of my experiments as a method for testing various babbitts for engineering purposes. For instance, engine-bearings for high tensions demand babbitts with high melting-points. Now, if a choice must be made from different brands of anti-friction metals, the latter having a smaller interval between their melting- and freezing-points, must be preferred as showing more homogeneity in the metal.—February 11th, 1906.

Royal Institution.—A General Monthly Meeting of the Members of the Royal Institution was held on the 5th inst., Sir James Crichton-Browne, M.D., F.R.S., Treasurer and Vice-President, in the Chair. Miss Ruddle Browne, Dr. G. L. Findlay, Miss M. H. Pamm, Mr. A. Sutton, Mr. L. C. Wallach, and Miss I. K. Young were elected Members. The Special Thanks of the Members were returned to Dr. Hugo Müller, F.R.S., for a donation of £100, to Professor J. A. Fleming, F.R.S., for a donation of £25, and to the Rev. J. H. Ellis, M.A., for a donation of £25 to the Fund for the Promotion of Experimental Research at Low Temperatures.

MEETINGS FOR THE WEEK.

MONDAY, 19th.—Society of Arts, 8. (Cantor Lectures). "Modern Warships," by Sir William White, F.R.S., &c.
TUESDAY, 20th.—Royal Institution, 8. "Food and Nutrition," by Prof. William Stirling, M.D., &c.
— Society of Arts, 8. "Illuminated Manuscripts," by H. Yates Thompson, F.S.A.
WEDNESDAY, 21st.—Society of Arts, 8. "The Fisheries of the North Sea," by Walter Garstang, M.A.
— Microscopical, 8. "Improved Method of taking Stereophotomicrographs and of Mounting the Prints," by H. Taverner. Exhibition of Orisidism, presented by N. D. F. Pearce.
THURSDAY, 22nd.—Royal Institution, 5. "The English Stage in the Eighteenth Century," by H. B. Irving, M.A.
FRIDAY, 23rd.—Royal Institution, 9. "The Internal Architecture of Metals," by Prof. J. O. Arnold.
SATURDAY, 24th.—Royal Institution, 3. "George Frederick Watts, as a Portrait Painter," by M. H. Spielmann.

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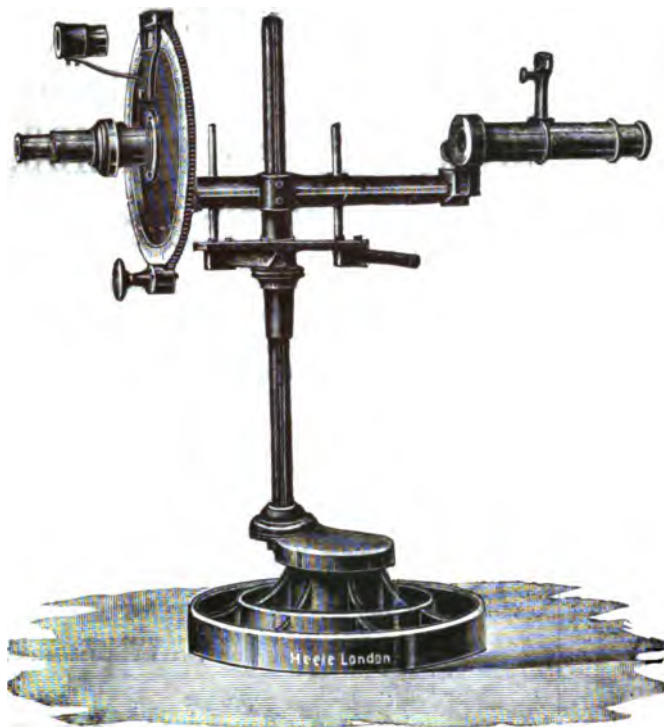
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AND
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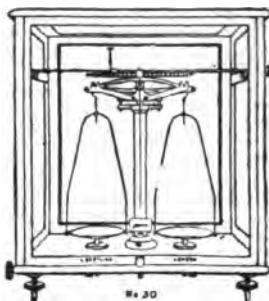
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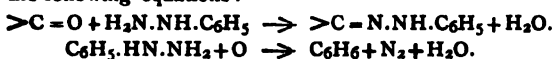
QUANTITATIVE DETERMINATION OF THE CARBONYL GROUP IN ALDEHYDES, KETONES, &c.

MODIFICATION OF THE STRACHE METHOD.

By WATSON SMITH, Jun.

IN seeking to minimise certain inconveniences in H. Strache's method of estimating quantitatively the carbonyl group,* it was found that the following modifications considerably improved the process.

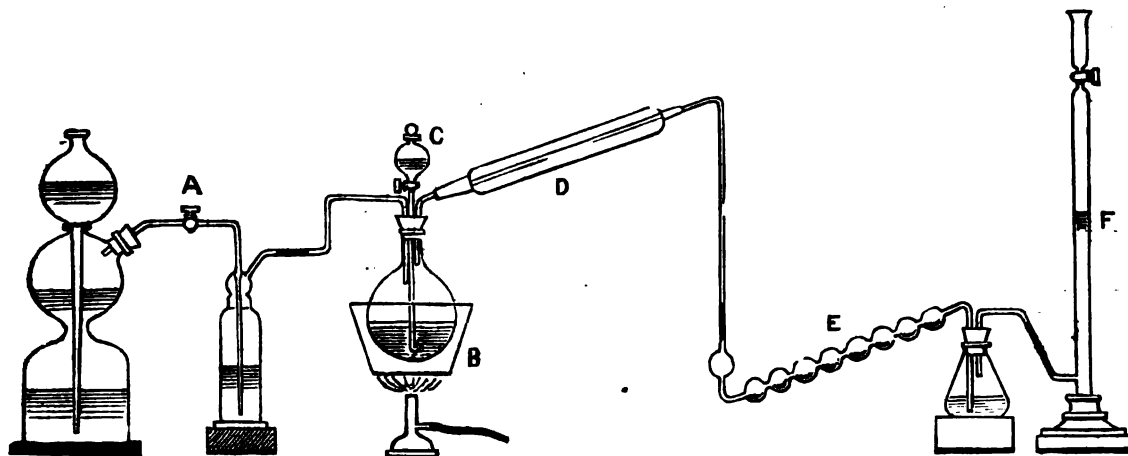
The method as to theory remains exactly the same, and consists in mixing the aldehyde or ketone with excess of phenylhydrazine; the excess is then oxidised by boiling with Fehling's solution, and the nitrogen gas liberated is collected and measured. The reactions are expressed in the following equations:—



The alterations in the process concern more especially the collection and estimation of the nitrogen gas.

The aldehydic or ketonic substance under examination (0.1 to 0.5 grm.) is mixed with an accurately measured quantity of the phenylhydrazine solution (1 part), and the sodium acetate solution (1½ parts) in a 100 c.c. measuring flask. Water is added to the mixture so as to make the volume up to about 50 c.c., and the liquid then heated on the water-bath for about fifteen minutes. After cooling, it is diluted to the 100 c.c. mark, well shaken, and the determination continued as follows:—

A stream of well washed carbon dioxide is led from the Kipp's apparatus into the flask B (see Fig.), which has a capacity of 750 to 1000 c.c., and contains 200 c.c. of Fehling's solution, on the surface of which floats a thin layer of paraffin oil. The tube which serves to lead the CO₂ into the flask does not dip below the surface of the liquid. The contents of the flask are heated, while a stream of CO₂ passes through the whole apparatus, until the bubbles of gas rising in the Schiff's gas-measuring apparatus are sufficiently small to be negligible. After the air has thus all been removed a blank experiment of the phenylhydrazine hydrochloride solution is made before the actual determination is begun in order to allow for this error. For this blank experiment, 10 c.c. of the solution are accurately measured out with the requisite quantity of sodium acetate solution, diluted to 100 c.c. and 50 c.c. pipetted into the dropping funnel c; the end of this is drawn out into a hook-shaped capillary, and dips down to the bottom of the flask, the object being to prevent the collection of bubbles of gas; the stem of the funnel is first filled with water. The solution in the funnel is allowed now to mix with



1. Instead of steam a current of carbon dioxide is used to drive the evolved nitrogen gas into the measuring tube.
2. Protection of the current of carbon dioxide from the absorptive action of the Fehling's solution by means of a thin layer of paraffin oil floating on the surface of the solution.
3. Absorption of the benzene in a suitable reagent.
4. Collection of the nitrogen in an ordinary Schiff's apparatus.

The reagents used are the following, and are the same as those used by Strache:—

Fehling's Solution.—CuSO₄ solution (70 grms. CuSO₄.5H₂O in 1 litre water). Alkaline potassium tartrate (350 grms. of tartrate and 260 grms. of KOH in 1 litre of water).

Sodium Acetate.—Ten per cent solution.

Phenylhydrazine Hydrochloride.—Five per cent solution.

the Fehling's solution, the funnel being then rinsed out once or twice with hot water. Any liquid tending to distil over while the mixture in the flask is being heated is condensed, and runs back into the flask by the inclined condenser D. The gas evolved by the reaction between the Fehling's solution and phenylhydrazine is now caught up in the current of CO₂, and carried into the absorption apparatus E, where it is washed free from benzene vapour; thence it is driven into the Schiff's apparatus filled with KOH solution (1:1). As soon as the bubbles have become as small again as before, the Schiff's apparatus is disconnected, allowed to stand some time, the gas volume being then read off, and reduced to 0° C. and 760 m.m. in the ordinary way.

The actual determination is made as soon as possible after the completion of this blank experiment.

Each determination requires about one hour for completion.

Of the substances suitable for absorbing the benzene vapour, the following were experimented upon:—

Paraffin oil and wax, caoutchouc, sulphuric acid, and a

* H. Strache, *Monatsh. für Chemie*, xli., 514; xlii., 299; *Benedikt and Strache, Ibid.*, xiv., 270; Hans Meyer, "Anleitung zur Quantitativen Bestimmung der organischen Atomgruppen."

mixture of sulphuric and nitric acids (nitrating mixture). Of these the nitrating mixture was found to act most suitably and to give the best results, especially when used in a bulb-absorption apparatus (see Fig.).

This mixture is made by mixing the acids in molecular proportions. The gas, after passing through the bulb-tube, is washed free from acid fumes by passing it afterwards through water contained in some suitable washing flask.

Determination of the Carbonyl Group in Hydroxy-benzaldehyde.

Reagent used for absorption of benzene vapours.	CO per cent. Found.	CO per cent. Theory.
Sulphuric acid. . .	19.52	22.80
Paraffin oil . . .	23.64	
	23.83	
Nitrating mixture, H ₂ SO ₄ + HNO ₃ . .	22.87	
	23.70	
	23.14	
	23.40	

Determination of the Carbonyl Group in Para-nitro-benzaldehyde.

Nitrating mixture . . .	19.22	18.53
	18.06	
	17.79	
	17.47	
	17.88	

Calculation of Results (Blayk Experiment with Phenyl-hydrazine Solution).

Average of Results.—One c.c. of the solution evolved on oxidation with Fehling's solution 12.08 c.c. N at 760 m.m. and 0° C.

0.2686 grm. of the hydroxybenzaldehyde was mixed with 10 c.c. of the phenylhydrazine solution, and 15 c.c. of the sodium acetate solution added. After heating on the water-bath, the solution was made up to 100 c.c. with distilled water.

50 c.c. were used in the determination. 39.46 c.c. of nitrogen were evolved (measured and reduced to 0° C. and 760 m.m.).

(5 × 12.08) = 60.40 c.c. — 39.46 c.c. = 20.94 c.c. of N, which represents the volume of nitrogen equivalent to the phenylhydrazine used in forming a hydrazone with the aldehyde. Then—

$$28.08 \text{ grms. N} = 28.00 \text{ CO} = 0.001256 : x.$$

$$x = 0.001252.$$

Or 1 c.c. of nitrogen is equivalent to 0.001252 grm. of CO; 20.94 c.c. are equivalent to 0.02622 grm. of CO; or the percentage—

$$\text{CO} = \frac{0.02622 \times 100}{0.1343} = 19.52 \text{ per cent of CO.}$$

My best thanks are due to Dr. F. Käuffer, of the Zürich Polytechnikum, for his kindly help and advice.

Zürich, January 28, 1906.

Royal Institution.—On Thursday next, March 1, at Five o'clock, Mr. Francis Darwin will deliver the first of three lectures at the Royal Institution on the "Physiology of Plants"; and on Saturday, March 3, at Three o'clock, Professor J. J. Thomson commences a course of six lectures on "The Corpuscular Theory of Matter." The Friday Evening Discourse on March 2 will be delivered by Dr. R. Caton, the subject being "Hippocrates and the Newly Discovered Health Temple at Cos"; and on March 9, by Dr. R. Hutchison on "Some Dietetic Problems."

ANALYSIS OF ALLOYS OF COPPER.*

By JOHN M. WILSON,
Assistant Inspector of Engineering Material, U.S.N.

THE analysis of alloys of copper with other metals such as lead, tin, antimony, iron, aluminium, zinc, nickel, and phosphorus is not difficult, but requires time, patience, and a reasonable amount of care. It is not like the analysis of steel, in which the analyst may weigh out several suitable portions from which to separate a number of elements sought, as in the analysis of such alloys nearly all of the constituents must be obtained from a single quantity, and each, in its turn, must be separated completely from the solution before proceeding to the next. In some cases we may separate the elements into groups insoluble in certain reagents, after which we may work the several groups for the elements contained therein. In either instance care must be taken to insure complete separation and thorough washing, and where it is necessary solution and re-precipitation must be resorted to, in order to make the separation complete.

Before taking up the chemical portion of this question, I desire to emphasise the necessity of proper sampling of the materials under consideration. Alloys of copper and zinc, copper, zinc, and tin, and of lead and tin are particularly prone to separate into their constituent parts, especially when there is a difference in the specific gravity of these constituents, and for this reason great care must be used to obtain cuttings which represent all portions of the cast. This is easily accomplished, in sampling sheets, bars, and wire, by cutting the entire cross section, or boring entirely through the piece. In the case of complicated castings, however, it is not quite so easy of accomplishment.

The best method of obtaining cuttings for analysis is to clamp the piece in a shaping machine, setting the tool so that it cuts small granular pieces, the size of a pin's head. A milling tool in a drill press answers admirably, provided proper care is taken to insure absence of cuttings of any other material in the tool. An ordinary drill is not a good tool to use for this purpose, on account of the difficulty of properly regulating the pressure so as to obtain granular cuttings. Mr. Reddig, an Assistant Inspector of Hull Materials, U.S.N., stationed at Reading, has invented a drill which, as I understand it, is shaped much like a carpenter's bit, i.e., a gimlet point in the centre, from which, on one side, extends a nearly flat lip, and on the other a lip cut into saw-teeth, so that the drillings are powdered.

Considerable time may frequently be saved and a choice of method of analysis indicated, by making a qualitative analysis of the thoroughly mixed cuttings, noting the comparative proportions of lead, copper, tin, and the presence or absence of arsenic and antimony, as well as the presence or absence of manganese and phosphorus.

The removal of copper as metal and lead as peroxide simultaneously, from a nitric acid solution containing 5 per cent of its volume of concentrated acid, by the electric current expedites the analysis, and does away with the tedious and unsatisfactory washing of a precipitate of mixed sulphides of copper and lead. The former has a tendency to oxidise, with considerable rapidity, on exposure to the air, and dissolve in the wash, passing in solution through the filter, only to re-precipitate in the filtrate. This may be prevented to a large extent by keeping the filter full, until the washing is complete, before allowing it to drain.

Sulphide precipitates containing lead are best dried roasted in porcelain, then taken up in HNO₃, and electrolysed. In the analysis of white metals the sulphide precipitate carries iron, and may contain zinc and nickel, in which case the liquid remaining after the removal of the lead and copper is to be neutralised with NH₄OH, and the

* The Chemical Engineer, ii., No. 3.

iron and other metals precipitated as sulphides, filtered off, and separated as described later.

The elements determined in these alloys are given below:—

Tin, in ordinary analyses, is that portion of the alloy insoluble in HNO_3 ignited over a blast-lamp.

Exact determination of tin is made by electrolysis of a solution in $\text{H}_2\text{C}_2\text{O}_4$ containing $(\text{NH}_4)_2\text{C}_2\text{O}_4$, after saturation with H_2S to remove antimony, and driving off the H_2S by heat. A current of 5 c.c. of electrolytic gas per minute is used, and allowed to run over night, when the tin will be found on the negative electrode as a grey coating. This may contain a little iron, which should of course be estimated and deducted.

Copper is deposited by the electric current from a HNO_3 solution containing about 5 per cent free acid by a current giving 5 c.c. of electrolytic gas per minute, and allowed to run over-night. The electrode is washed in the following manner:—The beaker is removed, and in its place is put one containing water, which is allowed to remain five to ten minutes; this, in turn, is removed, and a second beaker of water is raised and lowered sixteen times, in order to completely submerge the electrodes. This beaker is then removed, the current shut off, and the electrode disconnected, and placed in a beaker of alcohol, after which it is rinsed with ether, and dried over the steam table, being supported by its stem on a block of wood 2 inches square, and held down by a similar block.

Lead is separated at the same time as the copper, and deposited on the positive electrode as PbO_2 . The coating adheres well when the amount of lead is below 4 per cent and 1 gm. of material is used. When in excess of that amount, it flakes off, and can not be readily handled. When this occurs I usually make another determination, using half the quantity, and reversing the electrodes as recommended by Herrick.

Sometimes, however, the analysis is repeated, using 1 gm., and after removing the tin, the solution is evaporated with H_2SO_4 , diluted with water, the PbSO_4 filtered on a Gooch felt, ignited, and weighed. The filtrate is then electrolysed, and the lead not precipitated as sulphate obtained as PbO_2 . At other times the alloy is dissolved in $\text{HNO}_3 + \text{HCl}$, evaporated to a syrup, chilled by stirring, and the PbCl_2 precipitated by alcohol. The PbCl_2 is then taken up in $\text{NH}_4\text{C}_2\text{H}_3\text{O}_2$, and the lead precipitated and weighed as PbCrO_4 . Any lead not precipitated is found as PbO_2 on electrolysis of the copper.

I have frequently found that lead is not completely removed from solution as PbSO_4 , even by addition of alcohol. My attention was first called to this fact while making some analyses of babbitt metals. I found lead as PbSO_4 0.15 per cent upon electrolysing the filtrates for copper. I found as PbO_2 0.12 per cent. In white metals containing as high as 40 per cent lead, I invariably find 0.10 to 0.12 per cent lead as PbO_2 on electrolysing for copper.

Iron is determined by titration of its sulphate with KMnO_4 , 1 c.c. = 0.002 gm. Fe. Alloys which contain no tin are dissolved in HNO_3 , diluted to 300 c.c., the iron precipitated with NH_4OH , filtered, washed free from copper with NH_4OH and hot water, the $\text{Fe}_2(\text{OH})_6$ taken up in dilute H_2SO_4 , the solution reduced by passing through a zinc column, and titrated with KMnO_4 . Alloys containing tin are dissolved in $\text{HNO}_3 + \text{HCl}$, evaporated, the HNO_3 displaced with HCl , the solution made up to convenient volume, the copper, lead, tin, &c., precipitated and filtered through a dry filter, and a measured portion taken from which the iron is precipitated and estimated.

Aluminum is determined by difference.

Manganese in small amounts is weighed as Mn_3O_4 in large quantity as $\text{Mn}_2\text{P}_2\text{O}_7$.

Zinc in small amounts as ZnO by roasting ZnS , in amounts above 2 per cent as $\text{Zn}_2\text{P}_2\text{O}_7$. The solution is made neutral to methyl-orange, boiled, and $\text{NaNH}_4\text{HPO}_4$ solution added. The whole is then vigorously stirred and set aside at 80–90° C. until the flocculent precipitate

becomes granular and settles well, when it is filtered on a weighed Gooch felt, washed three or four times with hot water, dried at 100° C., ignited over a blast-lamp with cover on tight, cooled, and weighed.

This method of determining zinc is neat, expeditious, and exact; the precipitate filters rapidly, requires a minimum of washing, and is easily handled. A 10 per cent $\text{NaNH}_4\text{HPO}_4$ solution (neutral to methyl-orange) is employed as a precipitant, using 1 c.c. per 1 per cent Zn supposed to be present.

Phosphorus is precipitated by magnesia mixture from the $(\text{NH}_4)_2\text{S}$ solution of the insoluble in HNO_3 , and, after purification, weighed as $\text{Mg}_2\text{P}_2\text{O}_7$.

Nickel in small amount is weighed as NiO , in large amount by electrolysis or titration with KCN solution.

The electrolysis is made in a sulphate solution containing considerable $(\text{NH}_4)_2\text{SO}_4$ and a large excess of NH_4OH . A current of 10 c.c. electrolytic gas per minute is used for one and a-half hours, after which the current is increased to 15 c.c. per minute for another hour and a-half, at the end of which time the nickel should all be on the negative electrode.

Antimony is weighed as sulphide after ignition in a current of CO_2 .

Arsenic is not looked for quantitatively and seldom qualitatively in my laboratory, although it is used to a considerable extent in the manufacture of brass, to increase its tensile strength. Had I the time, therefore, I would test every sample received for this element. The best method is undoubtedly that of distillation after Lundin, as described by Blair, weighing the arsenic as As_2S_3 on a Gooch felt.

I will now describe briefly some apparatus and reagents used in the work.

Electrodes used are plain platinum cylinders, made without seams, to which are attached flattened wires well rivetted. The foil used is heavy, the better to stand wear and tear. The large cylinder is 2 inches high and 1½ inches in diameter and the small one is 2 by ½ inches.

Sodium Sulphide.—Being unable to obtain any sodium sulphide in the market suitable for the purpose, I prepare it, according to Classen, by dissolving one pound of caustic soda in five pints of water, dividing the solution into two approximately equal portions, one of which is saturated with H_2S (purified by passing through water and two U-tubes loosely packed with absorbent cotton) until it no longer increases in volume. The solution is then filtered into the other half, and the whole saturated as before, resulting in a light yellow solution, which is filtered into a large evaporating dish and evaporated rapidly till a pellicle forms, when the hot liquid is poured into 2 oz. bottles, the glass-stoppers sealed with paraffin, and the whole lot kept in a cool dark place. The solution keeps indefinitely without change of colour.

Ammonium Phosphate.—Add NH_4OH in slight excess to a 10 per cent solution of the salt, boil vigorously until nearly free from NH_3 , filter to remove any precipitate, and allow to cool. Make neutral to methyl-orange by the cautious addition of P_2O_5 or HCl .

Ammonium Sulphide is made in a similar manner to sodium sulphide, using two volumes water to one of concentrated NH_4OH , and omitting the evaporation.

Ammonium Acetate.— NH_4OH 400 c.c. + water 480 c.c. Add $\text{HC}_2\text{H}_3\text{O}_2$ glacial 340 c.c. The solution should be slightly acid to litmus; if not, add acid cautiously until the solution turns blue litmus red.

Solvent for White Metals.— HCl concentrated 800 c.c., HNO_3 concentrated 200 c.c., water 1000 c.c., KCl 4 grms.

Manganese Bronze.

Weigh 1 gm. into a No. 1 Griffin beaker, add 5 c.c. concentrated HNO_3 , heat on the steam table until no more nitrous fumes come off, add 25 c.c. water, digest until the insoluble matter, if any, appears perfectly white, filter through a double 7 c.m. ashless filter, and wash with 10 per cent HNO_3 until free from copper. Add NH_4OH

o the filtrate until a clear solution results, and then concentrated HNO_3 until the solution is acid and of a good copper-green colour, free from any yellow tinge. Add 5 c.c. HNO_3 in excess, and electrolyse over-night with a current of 5 c.c. electrolytic gas per minute. In the morning make sure the copper is all out of the solution, and if so, wash, dry, and weigh the electrodes for copper and $\text{PbO}_2 + \text{MnO}_2$. Dissolve the $\text{PbO}_2 + \text{MnO}_2$ from the electrode in hot HCl (1 to 1), evaporate to dryness, moisten with HNO_3 , evaporate until nearly dry, add 5 c.c. HNO_3 and 5 c.c. water, boil, and add PbO_2 . Boil two minutes longer, and allow to settle. If any violet colour of permanganic acid develops, titrate with standard sodium arsenite solution, and calculate the manganese so found to MnO_2 . Deduct from the weight of $\text{PbO}_2 + \text{MnO}_2$ found, The remainder will be PbO_2 . Calculate to Pb.

Although most writers claim that manganese is precipitated by electricity I have never found the amount precipitated together with the PbO_2 to exceed 0.05 per cent even in bronzes containing 3 to 31 per cent Mn.

Transfer the liquid remaining after removal of copper and lead, together with the washings, to a No. 4 beaker, boil, precipitate the iron and alumina with NH_4OH , filter into a large flask, reserve filtrate, dissolve precipitate in HCl , and precipitate with NH_4OH . Repeat once more to insure complete separation of Mn + Zn from Fe and Al.

Unite the three filtrates, make nearly neutral, and add enough bromine to colour the solution. Pour down the sides of the flask, in order to mix as little as possible, 75—100 c.c. NH_4OH , and allow to stand in the cold until the bromine has nearly disappeared from the bottom of the flask. Warm the flask gently, and bring its contents to a boil. Continue boiling until the precipitated MnO_2 collects in flasks, allow to settle, filter through paper. Wash three times with hot water, reserve filtrate, dissolve precipitate in $\text{HCl} + \text{H}_2\text{SO}_3$, boil off SO_2 , make slightly ammoniacal, and re-precipitate the manganese as before.

Unite the filtrates, evaporate to about 400 c.c., cool, make just neutral to methyl-orange, boil, and add $\text{NaNH}_2\text{HPO}_4$ (1 c.c. per each 1 per cent Zn calculated). Stir, set in a warm place (80 to 90° C.) until the precipitate becomes granular and settles well, filter through weighed Gooch felt, wash three or four times with hot water, dry at 100° C., ignite over a blast, with cover on tight, cool, and weigh as $\text{Zn}_2\text{P}_2\text{O}_7$.

Weigh 5 grms. into a No. 4 beaker, add 5 c.c. concentrated HNO_3 + 25 c.c. concentrated HCl , evaporate to dryness on a steam-plate, moisten with HCl , and evaporate again. Add 20 c.c. HCl + 50 c.c. water, boil, filter into 500 c.c. graduated flask, wash twice alternately with hot HCl (1—1) and cold water, then several times with hot water, ignite, and weigh the residue. Test SiO_2 with $\text{HF} + \text{HNO}_3$.

Calculate to Si, which is usually about 0.03 per cent. Make filtrate up to mark, pour into a beaker, warm to 60 to 80° C., saturate with H_2S , and filter through a dry ribbed filter into a 500 c.c. graduate until 400 c.c. have run through. Reject the filter and precipitate, transfer the filtrate to a beaker, evaporate to small volume or to dryness, and oxidise with HNO_3 , and boiling. The volume should now be 100 to 150 c.c. Make three basic acetate separations, precipitate, ignite, and weigh as Mn_3O_4 . If large take up in $\text{HCl} + \text{H}_2\text{SO}_3$, NH_4OH , and, if the precipitate is small, wash well with hot water, ignite, and weigh as Mn_3O_4 . If large take up in $\text{HCl} + \text{H}_2\text{SO}_3$, evaporate to dryness, moisten with HCl , take up in hot water, add 10—20 c.c. $\text{NaNH}_2\text{HPO}_4$, boil, add NH_4OH until a precipitate forms, stir till this becomes silky, and then add NH_4OH , a drop at a time, stirring constantly until the liquid smells distinctly of NH_3 . Set aside two to three hours, filter on a weighed Gooch felt, wash with NH_4OH (1 to 3) containing NH_4NO_3 , dry on the steam-table, ignite, and weigh as $\text{Mn}_2\text{P}_2\text{O}_7$. Calculate to manganese.

Dissolve the iron and alumina precipitate in dilute HNO_3 , precipitate with NH_4OH in a volume of 350 c.c., and filter. Wash twice on the filter (it is not necessary to

wash much, as any NH_4NO_3 adhering to the precipitate assists in preventing reduction of Fe_2O_3 during ignition), and transfer precipitate and wet filter to a platinum crucible. Moisten the filter thoroughly with concentrated HNO_3 , dry on a steam-table, place over a Bunsen turned low, with the crucible cover on nearly tight, and raise the temperature slowly to the full heat of the Bunsen. Roast one hour, ignite over blast fifteen minutes, raising cover occasionally, and weigh before crucible is cold. Repeat ignition, and weighing until the weight is constant. The precipitate is $\text{Fe}_2\text{O}_3 + \text{Al}_2\text{O}_3$.

Fuse precipitate with eight to ten times its weight of $\text{K}_2\text{S}_2\text{O}_7$ for several hours at a temperature just sufficient to drive off a little SO_3 . Leach melt in water, and filter off, estimate and deduct a little SiO_2 . Reduce the filtrate and titrate the iron. Calculate to Fe_2O_3 and also to iron ($\text{Fe}_2\text{O}_3 + \text{Al}_2\text{O}_3$) - $\text{Fe}_2\text{O}_3 = \text{Al}_2\text{O}_3$. Calculate to aluminum.

(To be continued).

CONDENSATION COMPOUNDS OF CAMPHOR- OXALIC ACID AND AMINES.*

SEVENTH COMMUNICATION ON CAMPHOROXALIC DERIVATIVES.†

By J. BISHOP TINGLE, Ph.D., F.C.S.,

and
WILLIAM EDWIN HOFFMAN, Jun., Ph.D.

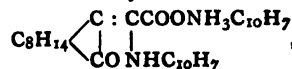
(Continued from p. 74).

ACTION OF AMINES ON CAMPHOROXALIC ACID.

I. β -Naphthylamine.

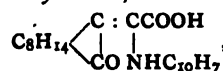
β -NAPHTHYLAMINE and camphoroxalic acid yield three compounds:—

1. The first of these, β -naphthylamine- β -naphthylcamphorformeneaminecarboxylate,—



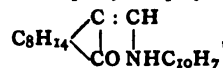
is formed in hot alcoholic solution and crystallises in slender pale yellow needles, melting at 160°.

2. The second compound is obtained when this salt, in very dilute alcoholic solution, is treated with sodium hydroxide and the product acidified. β -Naphthylcamphorformeneaminecarboxylic acid,—



crystallises from alcohol in slender bright yellow needles, melting at 172.5°. This compound has been previously described by J. Bishop Tingle and Alfred Tingle (*Trans. Am. Chem. Soc.*, 1901, xxiii., 375).

3. The third compound from β -naphthylamine and camphoroxalic acid, β -naphthylcamphorformeneamine,—



is formed by heating either of the preceding substances above its melting-point. It crystallises in slender pale yellow prisms, melting at 173°.

II. Paratoluidine.

Paratoluidine and camphoroxalic acid yield a series of three compounds, corresponding to those formed by β -naphthylamine.

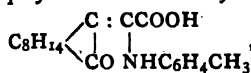
1. The p-toluidine p-tolylcamphorformeneaminecarboxylate,—



* From the *American Chemical Journal*, xxxiv., No. 3.

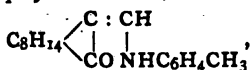
is formed in alcoholic solution, and crystallises readily from alcohol in pale yellow needles, melting at 152°.

2. *p-Tolylcamphoformeneaminocarboxylic acid*,—



is formed from the above salt by treatment with sodium hydroxide solution and subsequent acidification of the product. It crystallises from benzene in pale yellow prisms melting at 168°.

3. *p-Tolylcamphoformeneamine*,—

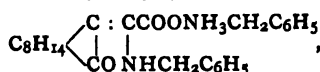


is formed by heating either of the two preceding substances above its melting-point. From alcohol it crystallises in slender yellow prisms melting at 178°.

III. *Benzylamine*.

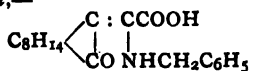
Benzylamine forms with camphoroxalic acid a series of three compounds, similar to those produced by β -naphthylamine and β -toluidine.

1. *Benzylamine benzylcamphoformeneaminocarboxylate*,—



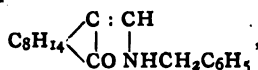
is obtained by the action of the free base on camphoroxalic acid, and crystallises from alcohol in irregular rhombohedra melting at 174.5°.

2. From the above salt, free *benzylcamphoformeneaminocarboxylic acid*,—



is formed by treating it with sodium hydroxide and acidifying the resulting solution. It is a colourless compound, crystallising from ethyl acetate in clusters of fine prisms, melting at 140°.

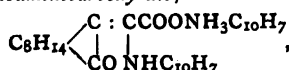
3. When heated above its melting-point, each of the preceding compounds decomposes, forming *benzylcamphoformeneamine*,—



which from alcohol crystallises in colourless prisms melting at 96.5°.

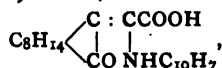
IV. *α -Naphthylamine*.

From α -naphthylamine and camphoroxalic acid two compounds are obtained; *α -naphthylamine naphthylcamphoformeneaminocarboxylate*,—



is formed in alcoholic solution, and, when purified by means of this solvent, yields greenish yellow prisms melting at 165°.

2. The above salt, when heated with sodium hydroxide solution and the product acidified, gives *α -naphthylcamphoformeneaminocarboxylic acid*,—



which crystallises from benzene with 0.5 molecule of benzene and melts at 170°.

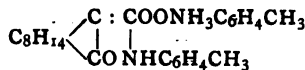
This acid has been described by J. Bishop Tingle and Alfred Tingle (*Journ. Am. Chem. Soc.*, 1901, xxiii., 375).

3. When heated above its melting-point, each of the two preceding substances formed a viscous mass, from which no definite compound could be isolated.

V. *Metatoluidine*.

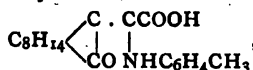
Metatoluidine and camphoroxalic acid also yield two compounds:—

1. *Metatoluidine metatolylcamphoformeneaminocarboxylate*,—



is formed when the free acid and amine react in alcoholic solution; it crystallises from alcohol in fine pale lemon coloured needles melting at 126°.

2. The above salt, when treated with sodium hydroxide solution and the product acidified, gives *metatolylcamphoformeneaminocarboxylic acid*,—

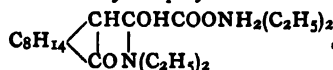


which crystallises from benzene in colourless needles and melts at 154°. When heated above its melting-point, each of the preceding substances formed a viscous mass, from which no definite compound could be isolated.

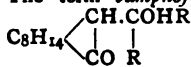
VI. *Diethylamine*.

Diethylamine and camphoroxalic acid react in alcoholic solution to form two compounds:—

1. *Diethylamine diethylcamphoformolaminocarboxylate*,—

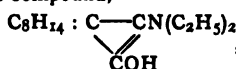


This compound crystallises from alcohol in colourless needles melting at 139.5°. It differs from the foregoing salts of camphoformeneaminocarboxylic acid in that its formation does not involve the elimination of the elements of water, in which respect it resembles the hydroxylamine derivative previously referred to (p. 72). With an alcoholic solution of ferric chloride no colour reaction is produced. The term *camphoformol* is suggested for the complex

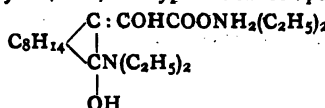


It differs from *camphoformene* by the addition of the elements of water, one hydrogen atom being united to the camphor nucleus.

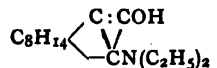
2. When this salt is heated above its melting-point it gives off water, carbonic anhydride, and diethylamine, and forms a compound of the same empirical formula as diethylcamphoformeneamine, $\text{C}_{15}\text{H}_{25}\text{NO}$, but which differs from what we should expect of that substance by virtue of the fact that, with ferric chloride and alcohol, it gives the reddish violet colour characteristic of the enol grouping; this seems to indicate that the reaction leading to its formation is hardly so simple as that which yields the corresponding derivatives of other amines. It may be possible that an isomeric compound,—



is produced, or it may be that diethylamine and camphoroxalic acid yield, first, the hypothetical compound,—



which, when heated, would doubtless give a body with the formula—

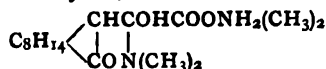


This matter will be investigated at the first suitable opportunity, because the substance in question might furnish an interesting case of dynamic isomerism.

The compound crystallises from ethyl acetate in colourless needles melting at 153°.

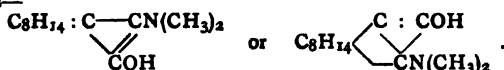
VII. Dimethylamine.

1. Dimethylamine and camphoroxalic acid in alcoholic solution react to form dimethylamine dimethylcamphorformaminocarboxylate,—



which crystallises from acetone in colourless needles melting at 137.5°. Like the corresponding compound from diethylamine, it is apparently formed by the direct combination of acid and base, without the loss of a molecule of water.

2. When the salt which has just been described is heated above the melting-point, it decomposes similarly to the analogous diethyl compound and forms a substance of the same empirical formula as dimethylcamphorformeneamine, $\text{C}_{13}\text{H}_{21}\text{NO}$, but which, on account of the fact that it gives a colour reaction with ferric chloride, does not seem to have that structure. It appears probable that its constitution is similar to that of the diethyl derivative, and that its formula is—



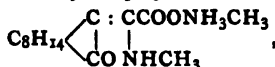
It crystallises from alcohol in colourless needles melting at 63°.

It is worthy of note that dimethylamine and diethylamine are the only two secondary amines from which it has been possible to obtain derivatives with camphoroxalic acid. Methylaniline and ethylaniline entirely fail to give definite compounds under similar conditions; whether this is due to the general tendency of these two substances to yield oily salts, or to their feeble basic properties as compared with the aliphatic amines, or to some other cause, must at present remain uncertain.

VIII. Methylamine.

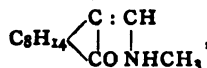
Methylamine forms two compounds with camphoroxalic acid:—

1. Methylamine methylcamphorformeneaminocarboxylate,—



which crystallises from alcohol in slender colourless needles melting at 172°.

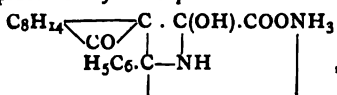
When this salt is heated above its melting-point, it forms methylcamphorformeneamine,—



which crystallises from ethyl acetate in colourless needles melting at 131°.

IX. Benzamidine.

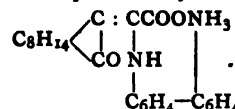
Camphoroxalic acid in alcoholic solution reacts with benzamidine in equimolecular proportion to form a colourless crystalline compound melting at 184°. At a slightly higher temperature it decomposes to a carbonaceous mass. Dilute acids and solutions of alkalis seem to have no effect upon the compound and ferric chloride in alcoholic solution does not give any colour reaction. The substance has the empirical formula $\text{C}_{19}\text{H}_{24}\text{O}_4\text{N}_2$. Its constitution may perhaps be represented by the expression—



but further work will be necessary to decide this point.

X. Benzidine.

Benzidine reacts with camphoroxalic acid in hot alcoholic solution, eliminating water and forming a greenish-yellow crystalline mass, which melts at 190° and decomposes when maintained at this temperature or heated for a short time at a higher one. The compound is apparently unchanged by the action of acids or alkalis and does not give any colouration with ferric chloride in alcoholic solution. In view of these facts and of the properties of benzidine itself, it is probable that one of the amino-groups has combined with the :COH group with the elimination of water, while the other has reacted with the carboxyl hydrogen to form a salt, as represented by the formula—



(To be continued).

PERKIN MEMORIAL AND JUBILEE OF THE COAL-TAR COLOUR INDUSTRY.

PUBLIC MEETING AT THE MANSION HOUSE.

THE year 1906 marks the fiftieth anniversary of the epoch-making discovery by William Henry Perkin of the dye-stuff "Mauve," by which the foundation was laid of the Coal Tar Colour Industry, and a great stimulus given to the study of organic chemistry.

The discoverer, Dr. Perkin, being fortunately still with us in full activity, it has been widely felt in this country and abroad that the jubilee of the foundation of this important industry should be made the occasion of a fitting public tribute to its founder, in recognition of the great services he has rendered to chemical industry and chemical science by his life work. We are also assured that the movement if inaugurated here would meet with the warmest support in Germany, where the seed first sown by Perkin has grown into an industry of national importance worth many millions a year. Those connected with this industry in Germany, we are led to believe, would welcome the opportunity of according to its founder some mark of appreciation and gratitude. It is also probable that in other countries we may count upon a generous measure of support.

With the object of considering what steps should be taken to give effect to the suggested memorial and jubilee celebration, a public meeting will be held at the Mansion House, London, on Monday, February 26th, at 3 p.m., at which the Lord Mayor has kindly consented to preside.

The meeting will be asked to recommend the opening of a subscription list for effecting the following objects:—

1. The presentation to Dr. Perkin for his lifetime of an oil portrait of himself, executed by an eminent artist, the portrait to become the property of the nation at his death.
2. The execution of a marble bust of Dr. Perkin to be placed in the rooms of the Chemical Society.
3. The establishment of a "Perkin Research Fund" for the promotion of chemical research, to be administered through the Chemical Society.

A strongly representative Provisional Committee of supporters of the scheme has been formed under the chairmanship of Prof. R. MELDOLA, F.R.S., President of the Chemical Society, which includes many important public men, scientists, and manufacturers.

Acyclic β -Chlorethyl and Vinyl Ketones.—E. E. Blaise and M. Maire.—The vinyl ketones can be prepared without much difficulty, and with satisfactory yields from the β -chlorethyl ketones. It is only necessary to boil the latter with diethylaniline under suitable conditions, $\text{CH}_2\text{Cl}-\text{CH}_2-\text{CO}-\text{R} \rightarrow \text{CH}_2=\text{CH}-\text{CO}-\text{R}$.—*Comptes Rendus*, cxlii., No. 4.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, February 1st, 1906.

Prof. R. MELDOLA, F.R.S., President, in the Chair.

Concluded from p. 78j.

*23. "The Relation between Absorption Spectra and Chemical Constitution. Part II. The Quinones and α -Diketones." By EDWARD CHARLES CYRIL BALY and ALFRED WALTER STEWART.

In the preceding communication, it was shown that in the absorption spectrum of ethyl pyruvate an absorption band is developed in a different region and at a different dilution from the bands given by the substances having a labile hydrogen atom (Baly and Desch, *Trans.*, 1904, lxxxv., 1029; 1905, lxxxvii., 766). This band was shown to be due to the two carbonyl groups in juxtaposition to one another; when two such carbonyl groups are present in the molecule, a new type of oscillation is induced, resulting in the absorption of light of a much longer wave-length than is absorbed by the process of enol-ketotautomerism. For this new type of oscillation, the name isorropesis was proposed. In the present paper it was shown that this isorropesis in α -diketones results in the absorption of light in the visible blue region, so that the substances are intensely yellow. This is evidenced by camphorquinone and diacetyl. An analogous condition in the close proximity of the carbonyl groups occurs in benzoquinone. It is shown that quinone develops the same absorption band as do diacetyl and camphorquinone. The process of isorropesis occurs therefore in benzoquinone exactly as in the case of the α -diketones, and is the origin of the yellow colour of this substance. The same is found to be true in the case of p -xyloquinone, toluquinone, α -naphthaquinone, acenaphthenequinone, and phenanthraquinone. There is little doubt, therefore, that the yellow colour of the quinones is due to the isorropesis between the carbonyl groups in juxtaposition. These observations strongly support Armstrong's theory that the colour of certain benzene derivatives is due to the quinonoid linking, for they show that the colour is caused, not directly by this linking, but by the isorropesis between the unsaturated atoms where this linking exists.

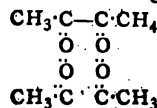
*24. "The Relation between Absorption Spectra and Chemical Constitution. Part III. The Nitranilines and the Nitrophenols." By EDWARD CHARLES CYRIL BALY, WALTER HENRY EDWARDS, and ALFRED WALTER STEWART.

In this paper are described the absorption spectra of compounds having the quinonoid linking and containing a nitrogen atom in place of one or both of the quinone oxygen atoms. By a comparison of the absorption spectra of the nitranilines with those of their hydrochlorides, it is shown that the free substances must exist in the quinonoid form, and that, as they show the same absorption band as benzoquinone and the α -diketones, a similar process of isorropesis takes place between the unsaturated nitrogen atoms as between the oxygen atoms of benzoquinone and the α -diketones. Similar arguments are found to hold good for the nitrophenols and for nitrosophenol. By these observations the principle developed in the preceding papers, that the colour of the quinones is due to the oscillation or isorropesis between the oxygen atoms, is extended to those quinonoid compounds containing nitrogen atoms in the place of one or both of the oxygen atoms.

DISCUSSION.

Prof. ARMSTRONG said that Mr. Baly had put aside entirely the view which had long been held that ketonic interactions were conditioned by the combination of various substances with the carbonyl group, and had adopted an entirely *intra*-molecular view of change, whether chemical or physical. He, however, was inclined

to advocate the view that the changes were *inter*-molecular. He also still adhered to the opinion that three absorbing centres were required to produce visible colour, *i.e.*, that iodoform, not methylene iodide, might be taken as typical of coloured substances. From this point of view, the colour of compounds such as diacetyl might be accounted for on the assumption that polymeric molecules were present, formed by the association, through the residual affinity of the oxygen atoms, of the ketonic groups, *e.g.* :—

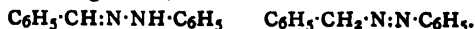


This explanation might perhaps apply also to metanitrophenol and metanitriline. He remarked subsequently that the blue colour of water might be accounted for from this point of view, but not by Mr. Baly's hypothesis.

In reply to Prof. Armstrong, Mr. BALY pointed out that the absorption spectrum of acetone and ethyl acetoacetate in aqueous solution shows that the tautomeric process is much decreased by the action of the solvent. Similarly, in the case of benzoquinone, the absorption band due to isorropesis exhibited in alcoholic solution entirely disappears when the substance is examined in aqueous solution, its place being taken by two bands in the ultra-violet region. These facts, due no doubt to the formation of hydrates, are strong evidence against the suggestion made by Prof. Armstrong, that the colour of diacetyl is due to a complex of two molecules of this substance.

*25. "The Action of Light on Benzaldehydophenylhydrazones." By FREDERICK DANIEL CHATTAWAY.

Fischer, who first prepared the ordinary stable α -modification of benzaldehydophenylhydrazone (*Annalen*, 1878, cxc., 135), noted that it reddened when exposed to the air, and this observation was confirmed by Biltz (*Zeit. Physik. Chem.*, 1899, xxx., 527), and Reutt and Pawlewski (*Bull. Acad. Sci. Cracow*, 1903, 503), who added that the reddening takes place on exposure to light, and disappears when the compound is heated or placed in the dark. The author has extended these observations, and shows that the presence or absence of air does not affect the coloration which is brought about by the violet and blue portions of the spectrum only. The changes of colour are probably due to a reversible isomeric change from the hydrazino- to the azo-configuration,—



The colour produced in benzaldehydophenylhydrazone when it has reached its maximum exactly equals in shade and intensity that of azobenzene itself. This explanation of the colour change is supported by the behaviour of benzaldehydediphenylhydrazone and benzaldehydebisphenylhydrazone, which are not capable of undergoing this type of change, and which are not altered in colour by light.

This intramolecular rearrangement caused by light and accompanied by colour change leads to the suggestion that the fading of organic colouring matters in sunlight may in some cases at least be due to a similar reversible isomeric change.

DISCUSSION.

Dr. WADE, who asked whether the author had noticed the production of the coloured modification of the hydrazone at the moment of its formation, more especially in benzene solution, stated that he had experienced great difficulty in obtaining a colourless product when the reacting substances were dissolved in this solvent.

Mr. W. ROBERTSON pointed out that according to the Hantzsch-Werner hypothesis two stereoisomerides are possible; in the case, however, of the benzaldehydophenylhydrazones, three isomeric modifications are known, and it is highly probable that all three possess different constitutions. The significance of the colour of the substituted benzaldehydophenylhydrazones when taken in con-

junction with the red unstable "benzylidenephénylhydrazine," was explained and the opinion expressed that the nitrobenzaldehydephenylhydrazones were, in reality, stable azo-compounds.

26. "The Union of Chlorine and Hydrogen." By CHARLES HUTCHENS BURGESS and DAVID LEONARD CHAPMAN.

The authors have investigated the dependence of the character of the induction period on the conditions. The results obtained increased the difficulty of explaining the phenomenon in question by the theory of an intermediate compound. They also indicated that the effect must be due to some retarding impurity. The impurity was found to be a gaseous compound resulting from the interaction of chlorine and ammonia. An inhibitive compound can also be formed by the action of chlorine on compounds capable of yielding ammonia on oxidation. When such substances are present in the actinometer, the retarding impurity can be gradually formed for considerable periods of time, so that the mixture after it has been previously induced can again become inert when it is allowed to remain in the dark. If the gases and the water contained in the actinometer are carefully purified from all ammoniacal impurity, no induction period can be observed even after the actinometer has stood for weeks in the dark.

Ammonia (chlorine-substituted ammonia) retards the action between chlorine and formaldehyde in the light. Its inhibitive effect is, however, much less in this case.

No difference could be detected in the absorption coefficients of mixtures in equal volumes of air and chlorine and of hydrogen and chlorine. The energy which brings about the combination of hydrogen and chlorine is therefore probably derived from the light absorbed by the chlorine in virtue of its optical properties.

27. "Note on the Molecular Weight of Epinephrine." By GEORGE BARGER and ARTHUR JAMES EWINS.

Abel and Taveau have recently (*Journ. Biol. Chem.* 1905, i., 1) objected to the formula $C_9H_{13}O_3N$ - 183 for epinephrine (adrenaline), the active principle of the adrenal gland. They prefer the formula $2(C_{10}H_{13}O_3N \cdot \frac{1}{2}H_2O) = 408$, and criticise the molecular weight determinations of previous investigators, arguing that the use of acetic acid is "entirely inadmissible," since the substance cannot be recovered unchanged from the solution. Von Firth (*Monatsh.*, 1903, xxiv., 261) has shown that at least part of the substance can be obtained crystalline by ammonia after evaporating the acetic acid in a vacuum, and the authors have confirmed this observation.

That the loss of epinephrine is extremely small in acetic acid solution was shown by Dr. H. H. Dale, who, using a physiological method indicated by Elliott (*Journ. Physiol.*, 1905, xxxii., 447), finds it possible to estimate epinephrine with an accuracy of about 5 per cent. This physiological method also served as a criterion for the purity of the epinephrine employed, the most active specimen obtained by repeated fractional precipitation with sodium carbonate and ammonia being selected.

The microscopic method of molecular weight determination (*Trans.*, 1905, lxxxvii., 1756) was employed, with glacial acetic acid and with benzaldehyde, at a temperature of 90°.

I. 0.0732 grm. in 2.00 grms. of acetic acid was intermediate between 0.19 and 0.214 grm.-molecule of benzil per 1000 grms. of solvent. $M = 172-193$.

II. 0.1016 grm. in 2.00 grms. of acetic acid was intermediate between 0.166 and 0.18 grm.-molecule of acetanilide per 1000 grms. of solvent. $M = 180-194$.

III. 0.0732 grm. in 2.00 grms. of acetic acid was intermediate between 0.205 and 0.21 grm.-molecule of benzil per 1000 grms. of solvent. $M = 174-179$.

IV. 0.0732 grm. in 2.00 grms. of benzaldehyde was intermediate between 0.21 and 0.22 grm.-molecule of benzil per 1000 grms. of solvent. $M = 167-174$.

The mean of all these values is 179.

The epinephrine solutions in Experiments II. and III. were heated to 90° for ten minutes (the time during which the capillary tubes were heated), then diluted with water, nearly neutralised, and made up to 1 in 10,000. On comparison with a solution of the same strength, made by dissolving the solid epinephrine in the minimum quantity of hydrochloric acid, Dr. Dale could not trace any loss of physiological activity.

The value obtained by Jowett (*Trans.*, 1904, lxxxv., 194) in a very dilute glacial acetic acid solution by the freezing-point method was 135, and by Bertrand (*Comptes Rendus*, 1904, cxxxix., 502) was 174.3. It would appear, therefore, that Abel's formula should be abandoned.

28. "The Critical Temperature and Value of ML/θ of some Carbon Compounds." By JAMES CAMPBELL BROWN.

The critical temperatures of a number of organic alcohols, acids, esters, and aromatic hydrocarbons are determined. The latent heats of the re-purified substances have been re-determined, and the values of the constant ML/θ are calculated for a number of these compounds for which this constant could not be given (*Trans.*, 1903, lxxxiii., 987, and *Trans.*, 1905, lxxxvii., 265). Two or three of the boiling-points are given more correctly for the purified substances.

The value of ML/θ rises very slightly with the increase of CH_2 in the aliphatic alcohols, acids and esters, but is very constant for the aromatic hydrocarbons.

(NOTE.—ML = molecular heat of vapourisation, θ = absolute temperature of the boiling-point.)

29. "Slow Oxidations in the presence of Moisture." By NORMAN SMITH.

Experiments have been carried out to determine whether the oxidation of ammonia at the ordinary temperature in the presence of air and water could be brought about (1) by catalysis, (2) by induced oxidation. The catalysts used were ferric oxide, stannic oxide, manganese dioxide, lead peroxide, and platinum. Stannic oxide and manganese dioxide produced both nitrite and nitrate from the ammonia (0.05 m.grm. of nitrate for 5 grms. of oxide used); ferric oxide and lead peroxide gave only small amounts of nitrite and nitrate (0.01 m.grm. of nitrate), whilst platinum appeared to be without action. With platinum heated to a temperature just below dull redness, however, a very dilute solution of ammonia or ammonium salts was readily oxidised in presence of air. In the presence of copper or tin undergoing oxidation in air, ammonia was readily oxidised to nitrite and nitrate; zinc in some experiments gave positive, and in others negative results, whilst ferrous and manganous hydroxides showed only traces of nitrite or nitrate. In the cases where the formation of nitrate was brought about by induced oxidation, the amount of nitrate produced was much greater than with catalysts.

The experiments were extended to the case of nitrogen. No evidence of oxidation of nitrogen by catalysts was obtained. In the presence of substances undergoing oxidation, the metals, zinc, iron, and magnesium only among a large number of substances investigated gave ammonia, nitrite, or nitrate. These results were shown to be probably due to traces of nitride in the metals.

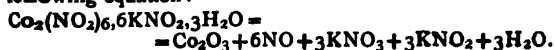
Water was evaporated in air, under various conditions, and it was shown that in no case was ammonium nitrite produced (compare Schönhein, *Journ. Prakt. Chem.*, 1861, lxxxiv., 215, and others) if the following sources of error were avoided:—(a) Traces of nitrite in the air, (b) nitrites produced in the burning of flames, (c) presence of ammonium compounds or albuminoid matter in the water, (d) traces of nitride in the metals.

No hydrogen peroxide was produced in the evaporation of water under various conditions, except in presence of certain metals such as zinc, which undergo rapid oxidation at the same time.

30. "Fischer's Salt and its Decomposition by Heat." By PRAFULLA CHANDRA RAY.

Fischer's salt as ordinarily prepared seems to correspond to the formula $Co_2(NO_2)_6 \cdot 6KNO_2 \cdot 3H_2O$. It is, however, invariably contaminated with appreciable traces of an oxide

of cobalt. The method of Rosenheim and Koppel evidently does not yield a purer product as claimed by them. The salt when heated in a vacuum decomposes according to the following equation:—

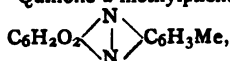


On this decomposition is based an accurate and expeditious method of analysing the compound.

31. "Action of Quinones on o-Diamines, o-Nitroaniline, m-Nitroaniline, and 2-Nitro-p-toluidine." (A Preliminary Note). By JAMES LEICESTER.

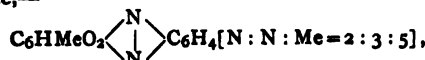
In a previous paper (*Ber.*, 1890, xxiii., 2793; *Abstr.*, 1890, 1445), it was shown that the action of o-nitroaniline and 2-nitro-p-toluidine on benzoquinone, toluquinone, and naphthaquinone was the same in principle as that occurring in the case of aniline and benzoquinone. Quinone-fluorindines and quinonephenazine derivatives were obtained on reduction with ammonium sulphide (*Proc.*, 1893, ix., 9).

Homofluorindine, $\text{C}_6\text{H}_4 \begin{smallmatrix} \text{NH} \\ \diagup \quad \diagdown \\ \text{N} \end{smallmatrix} \text{C}_6\text{H}_4 \begin{smallmatrix} \text{N} \\ \diagdown \quad \diagup \\ \text{NH} \end{smallmatrix} \text{C}_6\text{H}_4$, has now been prepared from quinonehomofluorindine, $\text{C}_6\text{H}_4 \begin{smallmatrix} \text{NH} \\ \diagup \quad \diagdown \\ \text{N} \end{smallmatrix} \text{C}_6\text{O}_2 \begin{smallmatrix} \text{N} \\ \diagdown \quad \diagup \\ \text{NH} \end{smallmatrix} \text{C}_6\text{H}_4$, by reduction with hydriodic acid (O. Fischer and E. Hepp, *Ber.*, 1890, xxxiii., 2789, and *Ber.*, 1888, xxi., 683). Various salts of the base have been made. Quinone- α -methylphenazine,—

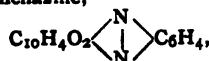


has also been reduced in a similar way. Some of the salts dissolve in alcohol with a deep blue colour, and have a red fluorescence; the slightest trace of ammonia changes the colour to bright red with a strong fluorescence.

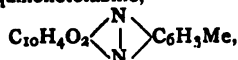
The absorption spectra of some of the compounds are under investigation with a view to the question of the origin of colour in organic compounds. Quinonephenotolazine,—



naphthaquinonephenazine,—



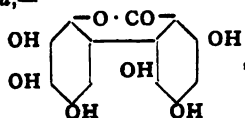
and α -naphthaquinonetolazine,—



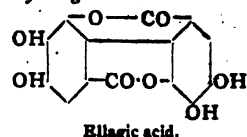
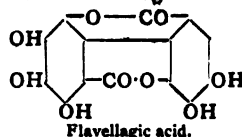
are also being examined in a similar manner.

32. "Some Oxidation Products of the Hydroxybenzoic Acids." (II.). By ARTHUR GEORGE PERKIN.

When gallic acid dissolved in 76 per cent sulphuric acid is oxidised by means of potassium persulphate, a colouring matter very similar to ellagic acid is produced. This substance, $\text{C}_{14}\text{H}_6\text{O}_9$, to which the name *flavellagic acid* is assigned, is obtained in yellow needles (m. p. above 360°), and dissolves in alkalis with a greenish yellow coloration, and gives an *acetyl* compound, $\text{C}_{14}\text{H}_9\text{O}_9(\text{C}_2\text{H}_3\text{O})_5$ (colourless needles, m. p. $317-319^\circ$), and a *benzoyl* derivative, $\text{C}_{14}\text{H}_9\text{O}_9(\text{C}_7\text{H}_5\text{O})_5$ (prismatic needles, m. p. $287-289^\circ$). On distillation with zinc dust, it gives *fluorene*, and on digestion with boiling potassium hydroxide solution is converted into a substance, $\text{C}_{13}\text{H}_8\text{O}_8$ (m. p. above 360°), the alkaline solution of which develops a bluish violet colour on oxidation. It crystallises with $1\text{H}_2\text{O}$ in almost colourless needles, gives an *acetyl* compound, $\text{C}_{13}\text{H}_8\text{O}_8(\text{C}_2\text{H}_3\text{O})_6$ (prismatic needles, m. p. $232-234^\circ$), and a corresponding *benzoyl* derivative (m. p. $261-263^\circ$). The results indicate that this substance is *hexahydroxydiphenylmethylolide*,—



and that flavellagic acid is *hydroxyellagic acid*—



When gallic acid is oxidised in acetic acid by means of potassium persulphate and sulphuric acid, *ellagic acid* is formed, and this process is preferable to that in which concentrated sulphuric acid is employed as the solvent (*Proc.*, 1905, xxi., 185), owing to the simultaneous production in this case of a small quantity of flavellagic acid. These colouring matters could only be separated in the form of their benzoyl derivatives.

33. "Contributions to the Chemistry of Oxygen Compounds. Part I. The Compounds of Tertiary Phosphine Oxides with Acids and Salts." By ROBERT HOWSON PICKARD and JOSEPH KENYON.

Tertiary phosphine oxides are easily prepared by the action of phosphorus oxychloride on organo-magnesium compounds. They combine with acids, metallic salts, and organo-magnesium iodides, forming compounds having the general formulæ $(\text{R}_3\text{PO})_2\text{H}_n\text{X}$, $(\text{R}_3\text{PO})_2\text{M}'\text{X}_2$, and $(\text{R}_3\text{PO})_2\text{CH}_3\text{MgI}$. The compounds described include some of those in which R is methyl, ethyl, propyl, phenyl, or benzyl, HX is ferrocyanic, chlorauric, cobalticyanic, dichromic, camphoric, iodobismuthic, or iodomercuric acids, and $\text{M}'\text{X}_2$ is zinc chloride or iodide, cadmium iodide, mercuric or cobalt chlorides. Other compounds of the oxides which do not conform to these general formulæ are those with hydrochloric, trichloroacetic, pyruvic, and chloroplatinic acids and cupric chlorides. The constitution of these compounds is best explained by Werner's theory of oxonium compounds (*Annalen*, 1902, cccxxxiii., 296). The oxides are very weak bases. Their aqueous solutions do not affect the birotation of dextrose, and their compounds with acids are hydrolysed to a large extent by water.

34. "The Rapid Electro-analysis of Metals." (Preliminary Note). By HENRY JULIUS SALOMON SAND.

A method for the rapid electro-deposition of metals for analysis and their separation by graded potential has been elaborated. Silver has thus been accurately separated from copper in about six minutes from boiling acetate solutions, more than half a grm. being deposited without the use of an auxiliary electrode. Copper was separated from bismuth from a boiling tartrate solution (time, about eight minutes). An auxiliary electrode is necessary. The operation must be repeated once, as the first deposit contains traces of bismuth. Owing to the high stirring-efficiency of the electrodes, bismuth has been easily obtained in a perfectly adherent form from nitrate solutions, the time required being about ten minutes, with no auxiliary electrode. By treating the precipitation of bismuth as a separation from hydrogen, perfectly adherent metallic deposits have also been obtained from tartrate and acetate solutions (with the use of an auxiliary electrode). So far the deposition of the following metals has been studied and successfully carried out:—Copper with currents of 10 amperes at about three volts, lead as peroxide, silver from boiling ammoniacal and acetate solutions, and bismuth.

PHYSICAL SOCIETY.

Annual General Meeting, February 9th, 1906.

Prof. J. H. POYNTING, F.R.S., President, in the Chair.

THE Reports of the Council and the Treasurer were read and adopted.

Report of the Council.

Since the last Annual General Meeting eleven ordinary Science Meetings and two informal meetings of the Society have been held. Of these eleven were held at the Royal

College of Science; one, that on March 24th, was held at University College, and one, on May 26th, at the National Physical Laboratory. The average attendance at the meetings, omitting the informal meeting at the National Physical Laboratory, when no record of the attendance was taken, has been 48.

During the past year the Council departed from the usual programme in setting apart one evening exclusively to an exhibition of apparatus. This innovation was well supported, manufacturers showing a readiness to exhibit, and the attendance of Fellows and visitors was greater than was anticipated, amounting to 240.

The number of Fellows now on the roll is 427, an increase of 3 over the number last year. Fourteen new Fellows have been elected. There have been eight resignations, and the Society has to mourn the loss by death of four Fellows, namely, W. Ackroyd, Rev. H. P. Gurney, Frank McClean, and H. R. Noble.

The number of students now on the roll is 4, the same number as last year. Two new students have been elected. There has been one transfer, and one resignation.

Report of the Treasurer for the Year 1905.

It has been increasingly difficult during the past two or three years to collect arrears of subscriptions, which now stand at a higher figure than usual, presumably in consequence of general depression and excessive taxation. With this exception the position of the Society remains practically unchanged. There is no unusual expenditure to report, except for binding periodicals and for refreshments at the soir  . At the present rate the Society is perhaps leaving rather too narrow a margin for contingencies. Allowing for liabilities the balance is about £10 less than last year, but we may hope that conditions will improve, before the balance is dangerously reduced.

Election of Honorary Fellows.

The following were elected as Honorary Fellows of the Society:—F. Kohlrausch, A. A. Michelson.

Election of Officers and Council.

The following Officers and Council were elected for the ensuing year:—

President—Prof. J. Perry, F.R.S.

Vice-Presidents—Those who have filled the Office of President, together with C. Chree, LL.D., Sc.D., F.R.S.; H. M. Elder, M.A.; Prof. J. A. Fleming, M.A., D.Sc., F.R.S.; and J. Swinburne.

Secretaries—W. R. Cooper, M.A., and Prof. W. Cassie, M.A.

Foreign Secretary—Prof. S. P. Thompson, B.A., D.Sc., F.R.S.

Treasurer—Prof. H. L. Callendar, M.A., LL.D., F.R.S.

Librarian—W. Watson, D.Sc., F.R.S.

Other Members of Council—T. H. Blakesley, M.A.; A. Campbell, B.A.; W. B. Croft, M.A.; W. Duddell; J. A. Harker, D.Sc.; W. A. Price, M.A.; S. Skinner, M.A.; S. W. J. Smith, M.A.; W. Watson, D.Sc., F.R.S.; Prof. H. A. Wilson, M.A., D.Sc.

Prof. J. PERRY then took the chair and delivered an Address.

He said that in the early days of the Society, when he had the honour of acting as a Secretary, and when Guthrie and Foster, Kelvin and Fitzgerald were Presidents, no Presidential Addresses were delivered, and he questioned whether we were not overdoing the business of requiring general addresses, which must almost always have as their theme the progress of science. Seldom did we find in such addresses new accounts of important original work, and he felt the inappropriateness of such an Address in speaking before a Society whose Proceedings were more intense with original work of the best kind than any other Society known to him with the exception of the Royal Society. He thought that every young reader of a paper before a scientific society made the mistake of assuming that his audience knew a great deal of the subject so familiar to

himself, and hence his paper was not understood. Writers of books on Physics assume their readers to be all truly logical students; they use words properly in a technical sense, and forget that many of their readers may use them in the newspaper writer's sense. For example, take the expression "adiabatic expansion." There are people who insist on finding that Rankine, Maxwell, and all others of our most exact writers are not only inconsistent with one another in the use or the expression, but that each is inconsistent with himself. If a portion of fluid expands slowly without gain or loss of heat, we know the way in which its p , v , and t alter as it changes state; this was originally called "adiabatic expansion," and the term has become a technical term for that kind of alteration of p , v , and t , however it may occur. Steam or air may be throttled through a non-conducting reducing valve, but the expansion is not adiabatic; although there is no gain or loss of heat. Steam or air passing along a pipe with friction, if it can only be made to lose heat through the metal of the pipe at exactly the proper rate at every place, is expanding adiabatically. When it is assumed that steam or air flows without friction from a vessel through an orifice, it is said that the expansion is adiabatic although it is rapid.

Referring to the teaching of physics to students entering upon the engineering profession, the President remarked that such teaching was nearly always slipshod. Many men enter a science college at the age of eighteen or more, knowing nothing of physical science. In the case of a great percentage of such men, it is impossible that they should acquire the scientific habit of thought. It is because so much of this kind of material is dealt with, that much of our teaching is slipshod. Every pupil entering a science college ought to have been experimenting and working graphically and numerically on physical science problems from a very early age, and then our science classes would deal with them in a scientific way. The causes of the unfitness of the average student are two:—One that his instincts and habits of thought were not trained from early youth; the other that his teachers in science colleges have absurd and uninteresting courses of study for him. In physics we are dealing with ideas which are not familiar to young students, ideas which can only become familiar in the laboratory. For example, such a simple mathematical idea as that of a decimal cannot be given in elementary schools in less than five or six years, whereas one week of weighing and measuring would give young children familiarity and clear ideas about decimals. Numerous examples could be given to prove that the principles of physics cannot be understood unless there has been early experimental training, and this is the reason why the professors of science in colleges of university rank and the professors in technical colleges obtain such poor reward for their labours. Referring to the many hundreds who every year take science degrees at the universities, and the thousands who pass the London University Matriculation examination, Prof. Perry remarked that if that was the standard of excellence of those present, his Address could serve no useful purpose. Nothing ought to be compulsory in schools except the study of English and of Natural Science. The object of a matriculation examination is to test whether a student entering a college will be able to benefit by the course of study there. The only language which ought to be compulsory in the Science department of a University is English. A Professor of Science ought to be allowed to teach his students in the way that seems best to him, and he should examine his students himself. Hedge him round with rules and regulations framed by Boards of Studies; tie him down to a syllabus, and the work he will do might be much better, certainly much more cheaply, done by a grinder at low wages. There is no one general elementary course in physics which all students ought to take: neither by their previous training nor from the uses which they will make of the principles of physics are they fit to be taught together. What is wanted is more classes, more rooms, and more teachers.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

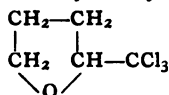
Comptes Rendus Hebdomadaires des Séances de l'Académie des Sciences. Vol. cxlii., No. 4, January 22, 1906.

Ebullition of Osmium, Ruthenium, Platinum, Iridium, Rhodium, and Palladium.—Henri Moissan.—All the metals belonging to the platinum family are easily melted and can be boiled in the author's electric furnace with currents varying from 500 to 700 ampères at pressure of 110 volts. Starting with 150 grms. of metal, fusion takes one or two minutes and regular ebullition is attained in about four minutes. Cold water is allowed to flow through a copper tube which is placed above the crucible in which the experiment takes place. The vapour is condensed on this in metallic drops formed of crystalline layers, each layer consisting of a network of crystals visible only under the microscope. All these metals when in the liquid state dissolve carbon; this latter substance crystallises out on cooling in the form of graphite. Osmium is the most difficult of all the metals to distil. Palladium is more easily fused than platinum, but is apparently less volatile than either platinum or rhodium.

Cathodic Phosphorescence of Europium.—G. Urbain.—The author has extracted europium from monazite sands, xenotime, and pitchblende. The earth prepared from any of these minerals by fractionation of the double magnesium nitrates in presence of excess of an isomorphous bismuth salt, shows a constancy of properties which is in general characteristic of definite elements. He now investigates the cathodic phosphorescence which europium excites when diluted to various degrees with other oxides.

Mixtures of Antimony and Tellurium, Antimony and Selenium, Cryoscopic Constancy of Antimony.—H. Pélabon.—Under the action of heat tellurium combines directly with antimony, forming a kind of mixture of the two bodies, consisting of antimony telluride accompanied by an excess of one or other of the elements. The mixtures thus obtained melt at temperatures below 620° and give homogeneous liquids, which, differing from antimony sulphide, do not separate into two superposed layers. The author investigates the solidification of these liquids and traces the curve of fusibility.

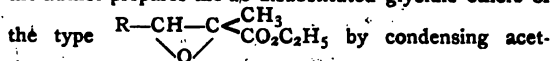
Methoxytrichloropentanol-1.5.4 and α -Trichloromethyltetrahydrofurfurane.—J. L. Hamonet.—Continuing his researches on the halogen ether oxides, $RO(CH_2)_nX$, the author investigates the reaction of anhydrous chloral on the magnesium derivative of iodomethoxypropane-1.3. The principal product of the reaction is methoxytrichloropentanol-1.5.4, $CH_3O.CH_2.CH_2.CH_2.CH.OH.CCl_3$. He examines the properties of this body and distils a mixture of it and phosphoric anhydride. The experiment is performed in a vacuum in a paraffin bath, and, by removal of a molecule of methanol, α -trichloromethyltetrahydrofurfurane,—



is obtained.

Acetylenic Amides and Nitrites.—Ch. Moureu and I. Lazennec.—In this preliminary note the authors give an account of the preparation and general properties of certain acetylenic amides. When hot, alcoholic potash saponifies these amides, giving the corresponding acetylenic acid, $R-C\equiv C-CO_2H$. By hydration, this is transformed more or less completely into β -ketonic acid, $R-CO-CH_2-CO_2H$, and then into the corresponding acetone, $R-CO-CH_3$, or acid, $R-CO_2H$.

Glycidic Condensation of Aldehydes with α -Chloropropionic Ether.—Georges Darzens.—In the fatty series, the author prepares the α -disubstituted glycidic ethers of



aldehyde, propylic aldehyde, and isovaleric aldehyde; but the yields are always very small (20 to 30 per cent at the most). Saponification of these ethers gives relatively stable acids. On the contrary the aromatic aldehydes give very good yields, and the glycidic acids thus prepared decompose sharply into carbon dioxide and ketones of the type $R-CH_2-CO-CH_3$.

NOTES AND QUERIES.

Examination Questions.—Can any reader recommend a book of Questions on Inorganic Chemistry suitable for revising before an examination? If possible, I should like one with a key, and comprising the whole subject as dealt with in such books as Shenstone's "Inorganic Chemistry," but I should very much like to know of one which comes as near to this as possible.—A. U.

MEETINGS FOR THE WEEK.

- MONDAY, 26th.—Society of Arts, 8. (Cantor Lectures). "Modern Warships," by Sir William White, F.R.S., &c.
TUESDAY, 27th.—Royal Institution, 5. "Food and Nutrition," by Prof. William Stirling, M.D. &c.
WEDNESDAY, 28th.—Society of Arts, 8. "London Traffic," by Capt. G. S. C. Swinton
THURSDAY, Mar. 1st.—Royal Institution, 5. "The Physiology of Plants," by Francis Darwin, M.A., &c.
Chemical, 8.30. "Studies of Dynamic Isomerism—Part IV., Stereoisomeric Halogen Derivatives of Camphor," by T. M. Lowry.
FRIDAY, 2nd.—Royal Institution, 9. "Hippocrates and the Newly Discovered Health Temple at Cos," by Richard Caton, M.D., &c.
SATURDAY, 3rd.—Royal Institution, 3. "The Corpuscular Theory of Matter," by Prof. J. J. Thomson, F.R.S., &c.



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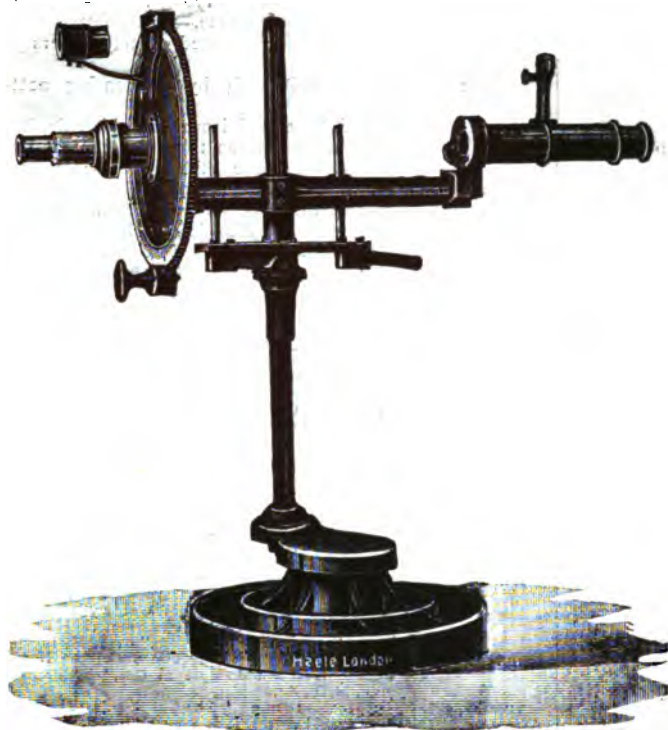
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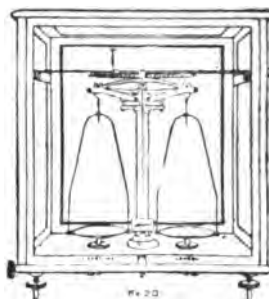
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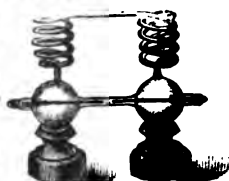
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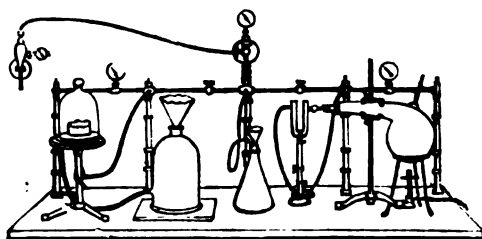
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THE CHEMICAL NEWS.

VOL. XCIII., No. 2414.

GALVANIC CELLS PRODUCED BY THE ACTION OF LIGHT.*

(SECOND COMMUNICATION).

THE CHEMICAL STATICS AND DYNAMICS OF REVERSIBLE AND IRREVERSIBLE SYSTEMS UNDER THE INFLUENCE OF LIGHT.

By MEYER WILDERMAN, Ph.D., B.Sc. (Oxon.).

The following is a summary of the different subjects dealt with in the paper:—

1. Further evidence is given that velocity of chemical reaction and chemical equilibrium in homogeneous systems follow under the action of light the laws of mass action.

2. Experimental proof that the E.M.F. produced by light in the different systems consists of two E.M.F.'s, viz., one created by light at a constant temperature due to the variation of chemical potential, and a thermo-E.M.F. simultaneously produced by the heating effect of the light, and due to the variation of the chemical potential with temperature (quantitative separation of the total E.M.F. into the two E.M.F.'s, and determination of the value of each E.M.F. in the different systems).

3. Experimental proof that the rays of all wave-lengths act both "chemically" and as "heat rays," only in different degrees.

4. Dealing with the experiments of Becquerel and Minchin, it is shown that phenomena observed by Becquerel and Minchin are not surface phenomena, but that their combination forms inconstant galvanic cells under the action of light. Experimental proof. Polarisation. (Influence of composition of the heterogeneous system upon its properties, as constant or inconstant galvanic cells. The course of the parts of the curves giving the induction and deduction periods in constant and inconstant cells).

5. On the nature of the chemical processes in galvanic cells created by light.

6. The method of investigation. The general arrangements of the experiments. The arrangements of a constant acetylene and arc light, of the photometer, the quartz vessel, the preparation of the plates, the bath, arrangements for calibration of the obtained results in standard units for photographing the effect of light upon different systems, &c.

7. On the E.M.F.'s in the dark, gas batteries, &c.

8. Chemical statics and dynamics of constant cells reversible in respect of the cation (e.g., Ag plates in NO_3Ag solution). The composition of such a system. The reactions going on in such a system. Proof that this system is reversible, that its composition remains constant under the action of the current, that the galvanic cells are *sui generis*. The relationship to Gibb's rule of phases.

9. Experimental results obtained with constant cells reversible in respect of the cation. The constant deflections in light. The course of the deduction and induction periods. The law of intensity. The E.M.F. obtained with the same system on extension of the experiments for longer periods. Influence of the composition of light upon the E.M.F. obtained (coloured screens). Influence of concentration of the solution upon the E.M.F. obtained.

10. The physico-mathematical theory of constant cells reversible in respect of the cation. The deduction of the general equation from the maximum work done by the system under the action of light (see *Roy. Soc. Proc.*, 1905). An experimental test and verification of the same in all its details. The E.M.F. and intensity of light. The

analogy between the action of heat and of light upon the systems, following from the direction of the current when the same plate is heated or illuminated (at a constant temperature); the author's "principle of movable equilibrium for light."

10. Chemical statics and dynamics of constant cells reversible in respect of the anion (e.g., Ag-BrAg plates in NaBr solution). The composition of such a system. The reactions going on in such a system. The mechanism of the reactions. Proof that such a system is reversible, that its composition remains constant under the action of the current, that they are *sui generis* (points of difference from ordinary galvanic cells). The relationship to Gibb's rule of phases.

12. Experimental results with constant cells reversible in respect of the anion, the course of the induction and deduction periods, the law of intensity, influence of composition of light (coloured screens). Acetylene and arc. Influence of concentration of the solution, influence of temperature, influence of the cation, transformation of constant cell into inconstant cells under the action of light, and vice versa, the rôle of the electrode in light cells. Experimental determination of the electrical potentials between the illuminated and not illuminated parts of the solution; what is to be understood under "the electrode reversible in respect of the anion"; on the conditions, under which constant reversible galvanic cell can be obtained, &c.

13. The physico-mathematical theory of constant cells reversible in respect of the anion. A detailed theoretical and experimental investigation similar to that in 10.

14. On the E.M.F. of constant reversible cells and the intensity of light (Experimental proof).

15. Chemical velocity of reaction in homogeneous systems when they are shifted to a new point of equilibrium by light at a constant temperature, follows, after the induction period has passed, the same law of mass action as in the dark. Further extensive experimental confirmation given by "galvanic cells created by light."

16. Chemical equilibrium in heterogeneous systems when shifted by light at a constant temperature to a new point, follow, after the induction period has passed, the same laws as in the dark.

17. The maximum work (containing the constant of equilibrium) and the law of intensity. Experimental proof that in homogeneous systems—

$$RT \ln \frac{C_1^{n_1} C_2^{n_2} \dots}{C_3^{n_3} C_4^{n_4} \dots} = RT \ln K = RT \ln \frac{k_1}{k_2} = C.I;$$

where k_1 , k_2 , are the velocity constants, K the constant of equilibrium.

18. The connection between the velocity of chemical reaction produced by the action of light, the intensity of light and absolute temperature; (a) when both reactions go on under the action of light only—

$RT \ln K_1 = C''I$ or $C'I + (k)$ and $RT \ln K_2 = C'''I$ or $C''I + (k)$,
(b) if only one reaction goes on in light, the other also in the dark,—

$$RT \ln k_1 = C''I + RT \ln k_2.$$

At a constant temperature—

$$\ln k_1 = C'v.I + K_{IV}.$$

19. The velocity of molecular or physical reactions between different parts of the heterogeneous system produced by and going on only under the action of light, follow the law found by the author for those reactions in the dark (*Report British Association*, Liverpool; *Zeit. Physik. Chem.*, 1899, vol. xxx., pp. 348—368).

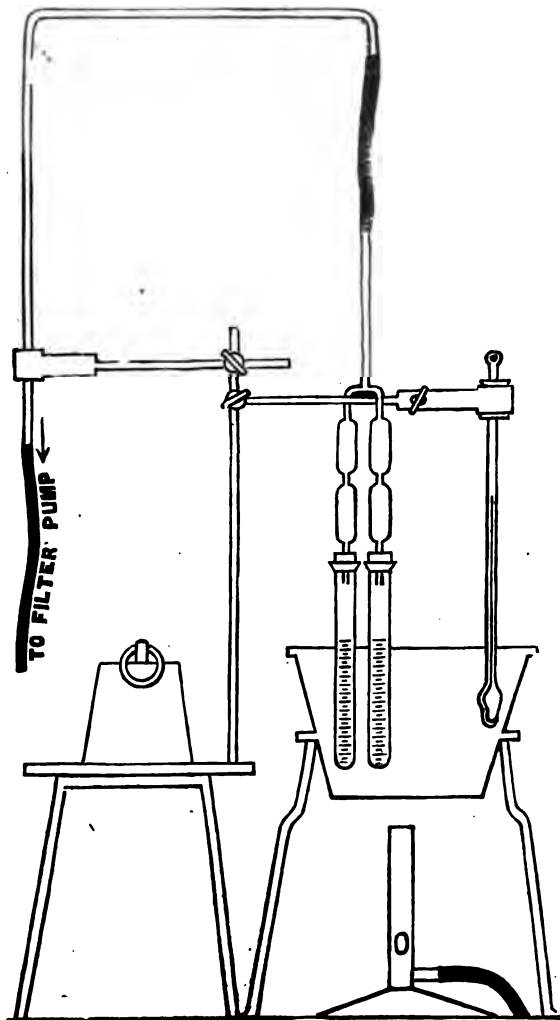
20. Velocity of chemical reaction in heterogeneous systems produced by and going on only under the action of light follow, after the induction period has passed, the laws deduced by the author for velocity of chemical reaction in heterogeneous systems in the dark (*Zeit. Physik. Chem.*, 1899, vol. xxx., pp. 371—382; *Phil. Mag.*, 1902, [6], pp. 468—489).

* Abstract of a Paper read before the Royal Society, January 25, 1906.

FURTHER EXPERIMENTS ON A NEW METHOD
OF DETERMINING MOLECULAR WEIGHTS.*

By PHILIP BLACKMAN.

It was found that, with solvents of fairly high boiling-points, no results could be obtained with the apparatus and method described by the author in *Journal of the Chemical Society*, 1905 (*Trans.*, lxxxvii., 1474), the boiling being extremely violent and irregular. The attempt was then made to carry out the operations under reduced pressure (this being effected by connecting the free end of the



T-piece with a filter-pump), and the results were much more satisfactory. It was found that most of the evaporated solvent, instead of escaping, condensed in the upper (cooler) portions of the apparatus, the condensed liquid remaining in the bulbs (the soft rubber tubing employed often collapsed, and this alone would have prevented any very considerable quantity of vapour escaping). In reading the volumes of the solutions care had to be taken that the solvent in the bulbs did not flow back into the test-tubes. No difficulty was met with in doing this,

for, on raising the apparatus from the water-bath, the excess of pressure within the test-tubes over that in the bulbs was quite sufficient to prevent the liquid in the bulbs from returning to the tubes for a considerable time.

Danger of ejection of the solution or of mixing, caused by any violent ebullition, will be obviated by having two bulbs instead of one in each of the limbs of the T-piece. The lower the boiling-point of the solvent employed, the easier will it be to carry out the determinations, and the more reliable will the results be.

A condenser should not be employed with the apparatus (compare *Trans.*, 1905, lxxxvii., 1476, Precaution 8), as not only will it destroy the mobility of the apparatus (as the latter would have to be firmly clamped to prevent it overturning), but it will also render it liable to break.

Experiments were carried out by conducting air at the same rate through the solutions, but the results were not satisfactory.

Results.

In the following experiments, a filter-pump and a water-bath were always used; also, instead of platinum wire, small pieces of broken glass (of inappreciable volume) were employed to assist in regulating the boiling.

After the first reading (No. 1), taken when a considerable quantity of liquid had collected in the bulbs, the liquid was allowed to flow back into the test-tubes. A second reading (No. 2) was then taken when a fair amount of liquid had again condensed in the bulbs. This process was repeated as often as was convenient.

- I. Compared *p*-dibrombenzene (m_1, v_1) with 1-chlor-2,4-dinitrobenzene (m_2, v_2). Equal weights of each (0.07 grm.); solvent, alcohol (96 per cent); time, thirty minutes; ebullition, steady.

$$m_1/m_2 = 236/202 = 1.168.$$

No.	v_1 C.c.	v_2 C.c.	v_1/v_2
1.	7.3	8.5	1.16
2.	7.0	8.2	1.17
3.	6.5	7.6	1.16
4.	6.0	7.0	1.16
5.	5.5	6.5	1.17

- II. Compared *p*-chloronitrobenzene (m_1, v_1) with benzil (m_2, v_2). Equal weights (0.08 grm. each); solvent, alcohol (96 per cent); time, thirty minutes; ebullition steady.

$$m_2/m_1 = 210/157 = 1.337.$$

No.	v_1	v_2	v_1/v_2
1.	8.5	6.5	1.31
2.	8.4	6.3	1.33
3.	8.0	6.0	1.33
4.	7.4	5.5	1.34
5.	7.1	5.2	1.34

- III. Compared *p*-nitraniline (m_1, v_1) with *p*-nitrobenzoic acid (m_2, v_2). Equal weights of each (0.07 grm.); solvent, alcohol (96 per cent); time, twenty minutes; ebullition regular.

$$m_2/m_1 = 167/138 = 1.21.$$

No.	v_1	v_2	v_1/v_2
1.	8.4	7.0	1.20
2.	8.3	6.8	1.22
3.	8.0	6.6	1.21
4.	7.5	6.1	1.23

- IV. Compared *p*-nitraniline (m_1, v_1) with hydroquinone (m_2, v_2). Equal weights (each 0.08 grm.); solvent, alcohol (96 per cent); time, thirty-five minutes; ebullition steady.

$$m_1/m_2 = 138/110 = 1.25.$$

* A Paper read before the Chemical Society, December 21, 1905.

No.	v_1 . C.c.	v_2 . C.c.	v_2/v_1 .
1.	6.8	8.2	1.21
2.	6.3	7.7	1.22
3.	5.7	7.3	1.28
4.	5.5	7.0	1.27
5.	5.0	6.3	1.26
6.	4.8	6.0	1.25

V. Compared *p*-nitrotoluene (m_1, v_1) with *p*-dibrombenzene (m_2, v_2). Equal weights of each (0.1 grm.); solvent, alcohol (96 per cent); time, twenty minutes; ebullition somewhat unsteady.

$$m_2/m_1 = 236/137 = 1.72.$$

No.	v_1 .	v_2 .	v_2/v_1 .
1.	6.5	3.7	1.75
2.	6.0	3.4	1.76
3.	5.4	3.1	1.74

VI. Compared phenolphthalein (m_1, v_1) with *p*-resorcylic acid (m_2, v_2). Equal weights (each 0.09 grm.); solvent, alcohol (96 per cent); time, twenty minutes; ebullition somewhat irregular.

$$m_1/m_2 = 318/154 = 2.06.$$

No.	v_1 .	v_2 .	v_2/v_1 .
1.	3.7	7.5	2.03
2.	3.3	7.0	2.12
3.	3.5	7.2	2.06

VII. Compared 1-brom-3.4-dinitrobenzene (m_1, v_1) with hydrazobenzene (m_2, v_2). Equal weights (0.07 grm. each); solvent, alcohol (96 per cent); time, twenty-five minutes; after fourth reading the boiling became violent.

$$m_1/m_2 = 247/184 = 1.34.$$

No.	v_1 .	v_2 .	v_2/v_1 .
1.	6.0	8.0	1.33
2.	5.8	7.8	1.34
3.	5.5	7.3	1.33
4.	5.2	7.0	1.34

VIII. Compared phenolphthalein (m_1, v_1) with *p*-cresotinic acid (m_2, v_2). Equal weights (each 0.1 grm.); solvent, alcohol (96 per cent); time, thirty minutes; ebullition regular.

$$m_1/m_2 = 318/152 = 2.09.$$

No.	v_1 .	v_2 .	v_2/v_1 .
1.	4.0	7.5	1.87
2.	3.5	6.9	1.97
3.	3.4	6.8	2.00
4.	2.8	5.8	2.07
5.	1.0	2.0	2.00

IX. Compared iodoform (m_1, v_1) with *m*-cresotinic acid (m_2, v_2). Equal weights of each (0.1 grm.); solvent, alcohol (96 per cent); time, twenty minutes; after third reading boiling became violent.

$$m_1/m_2 = 394/152 = 2.59.$$

No.	v_1 .	v_2 .	v_2/v_1 .
1.	3.6	8.0	2.22
2.	2.5	5.8	2.32
3.	3.0	7.5	2.50

X. Compared *p*-amidophenol (m_1, v_1) with α -naphthol (m_2, v_2). Equal weights (0.1 grm. each); solvent, alcohol (96 per cent); time, fifteen minutes; after second reading boiling became very violent.

$$m_2/m_1 = 144/109 = 1.32.$$

No.	v_1 .	v_2 .	v_2/v_1 .
1.	6.2	5.0	1.24
2.	6.0	4.8	1.25

XI. Compared 1-brom-3.4-dinitrobenzene (m_1, v_1) with *o*-cresotinic acid (m_2, v_2). Equal weights of each (0.07 grm.); solvent, benzene (boiling-point 80–82°); time, one hundred and seventy minutes; ebullition slightly irregular.

$$m_1/m_2 = 247/152 = 1.62.$$

No.	v_1 . C.c.	v_2 . C.c.	v_2/v_1 .
1.	5.5	7.5	1.36
2.	5.0	7.0	1.40
3.	3.5	6.0	1.71
4.	3.0	5.0	1.66
5.	5.0	8.0	1.60
6.	4.0	6.0	1.50
7.	4.0	6.2	1.55
8.	3.5	6.0	1.71
9.	3.0	5.2	1.73
10.	4.5	7.0	1.55
11.	3.5	6.0	1.71
12.	3.3	5.8	1.76
13.	3.5	6.0	1.71

XII. Compared *p*-cresotinic acid (m_1, v_1) with cinnamic acid (m_2, v_2). Equal weights of each (0.07 grm.); solvent, benzene (boiling-point 80–82°); time, twenty minutes; ebullition somewhat unsteady.

$$m_1/m_2 = 152/148 = 1.03.$$

Volumes v_1 and v_2 were nearly always equal, giving $v_2/v_1 = 1.0$ approximately.

XIII. Compared hydrazobenzene (m_1, v_1) with *m*-cresotinic acid (m_2, v_2). Equal weight of each (0.07 grm.); solvent, benzene (boiling-point 80–82°); time, forty minutes; ebullition somewhat irregular.

$$m_1/m_2 = 182/152 = 1.19.$$

No.	v_1 .	v_2 .	v_2/v_1 .
1.	6.0	7.3	1.21
2.	5.5	6.6	1.18
3.	4.5	5.8	1.29
4.	4.0	5.0	1.25
5.	3.7	4.5	1.21
6.	3.3	4.1	1.24
7.	3.0	3.6	1.20

XIV. Compared phenolphthalein (m_1, v_1) with diphenylamine (m_2, v_2). Equal weights of each (0.08 grm.); solvent, acetone (boiling-point 56.5–57°); time, forty-five minutes; ebullition steady.

$$m_1/m_2 = 318/169 = 1.88.$$

No.	v_1 .	v_2 .	v_2/v_1 .
1.	4.4	7.6	1.73
2.	3.8	7.2	1.89
3.	3.4	6.7	1.97
4.	1.8	3.4	1.88
5.	3.2	6.0	1.87
6.	2.7	5.1	1.88
7.	2.0	3.6	1.80
8.	2.2	3.8	1.73

XV. Compared *p*-dibrombenzene (m_1, v_1) with *p*-nitrotoluene (m_2, v_2). Equal weights of each (0.08 grm.); solvent, acetone (boiling-point 56.5–57°); time, forty minutes; ebullition steady.

$$m_1/m_2 = 236/137 = 1.72.$$

No.	v_1 .	v_2 .	v_2/v_1 .
1.	4.0	7.0	1.75
2.	3.7	6.7	1.81
3.	3.5	6.3	1.80
4.	3.3	6.0	1.82
5.	3.0	5.3	1.77
6.	2.5	4.5	1.80
7.	2.8	5.0	1.78

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THE RADIO-ACTIVITY OF THE URANYL
DOUBLE SALTS.

By W. MARCKWALD.

THE remarkably great radio-activity of certain uranyl double salts has recently been mentioned in several publications. R. J. Meyer and Fritz Wendel (*Berichte*, 1903, xxxvi., p. 4655) first called attention to the fact that potassium and ammonium uranyl nitrate "have powerful radio-active effects upon the photographic plate." E. Rimbach (*Berichte*, 1904, xxxvii., p. 461) went into this question rather more deeply. With H. Bürger and A. Grewe he investigated many uranyl double nitrates, chlorides, and sulphates with alkalis and other bases, and he found that most of the salts examined produced no blackening of the photographic plate after exposures of twelve hours. "But a strong effect was produced with the potassium and rubidium double sulphate, and most of all with the double nitrates of potassium, ammonium, and rubidium." Rimbach's fellow worker, A. Grewe, in his Dissertation "Beiträge zur Kenntniss der Doppelsalze im Unwandelungsintervall," Bonn, 1905, describes more extensive experiments on radio-activity, especially that of uranyl potassium nitrate, compared with uranyl nitrate. He compared the radio-activity of the two salts not only by means of the photographic plate, but also with an electroscope, and came to the conclusion that the activity of the salts in the latter case was of the same order of magnitude, but that, on the other hand, the double salt had a powerful effect upon the photographic plate in twenty-four hours, while the action of uranyl nitrate itself was hardly appreciable.

The fundamental experiments of H. Becquerel and of P. and S. Curie on the radio-activity of uranium and its compounds showed that in the many uranium compounds which they investigated the strength of the activity corresponds approximately to the amount of uranium the compound contains. This fact is one of the most important supports of Rutherford's theory of the cause and nature of radio-activity, which has proved so fruitful. It is clear that the foundations of our theoretical views would be destroyed if the assumption of the above authors should prove to be correct, that certain uranium compounds could possess a higher radio-activity than uranium itself.

For this reason it appeared of interest to investigate this question thoroughly. Dr. R. J. Meyer was kind enough to place his preparations at my disposal, for which I owe hearty thanks to him, and also to Dr. H. Grossman, who sent me two ethylene diamine double salts of uranyl which had not yet been described, namely, the sulphate and nitrate, the latter of which exhibited an abnormally strong action upon the photographic plate.

Besides these two ethylene salts, I have examined uranyl potassium nitrate, uranyl thallium nitrate, and a specimen of uranium nitrate used to prepare these salts. The activity of the salts was first determined quantitatively in the usual way by means of a sensitive electrometer, and was found to be of the same order of magnitude for all the salts. As the quantities of the salts at my disposal enabled me to spread them out over a surface of 16 sq. c.m. only, the measured saturation current was too small to allow the more minute differences to be detected. It amounted to about $0.2 \cdot 10^{-11}$ ampères. These measurements confirmed and supplemented Grewe's observations with the electroscope. Hence the α -radiation of all the salts examined is approximately the same as the theory requires.

The question now occurred whether the β -radiation would show considerable differences. The above mentioned authors, with the exception of Grewe, have not expressly stated that in their photo-chemical experiments they protected the sensitive plate from the action of light by means of opaque paper. Only Grewe mentions that the action of potassium uranyl nitrate on the photographic plate is not perceptibly lessened by means of opaque paper. This

latter statement, according to my experiments, is undoubtedly founded on an error.

A photographic plate was wrapped in opaque paper in such a way that there was a single layer of paper on the film side. On this pieces of glass, lead wire, and thin aluminium foil were distributed, and the five above mentioned salts contained in thin walled little glass vessels were laid directly on these. After twenty-four hours the plate was developed. With all the salts the action was uniformly very small, and scarcely perceptible. When the experiment was repeated under the same conditions, except that the plate was exposed for six days, it was distinctly blackened wherever the salts had lain. The pieces of glass and lead appeared as brighter images, while the aluminium, which is easily penetrated by β -rays, was more faintly indicated. In this case also the action of all five salts was about the same. Hence no considerable difference can be established in the β -radiation of the five salts.

The result was totally different if the salts in the little glass vessels were laid directly on the photographic plate. After twenty-four hours the plate appeared strongly blackened where the potassium uranyl nitrate and the ethylene diamine uranyl nitrate had been, while, on the other hand, the action of the uranyl nitrate, of the thallium uranyl nitrate, and ethylene diamine uranyl sulphate was distinct, but very much fainter. It follows from the foregoing that the effect is produced by the action of light rays but not Becquerel rays, and this was seen still more clearly when pieces of glass, opaque paper, and aluminium were laid on the photographic plate, and the glass vessels on these. The glass, which was 1.5 m.m. thick, was penetrated by the active rays without any diminution in strength, while on the contrary the paper and aluminium, which were 0.2 m.m. thick, threw sharp shadows.

This result thus shows that the above uranium salts emit light rays, and the potassium and ethylene diamine double nitrate to a much higher degree than the other salts. Both the double salts differ from the three others externally because of their strikingly beautiful fluorescence. This power of luminescence can be due to one of two causes. The most obvious explanation would be to suppose that these salts, like other phosphorescent substances, emit light for a long time after they have been illuminated. However, the photo-chemical action of these salts is not diminished even after they have been kept in the dark for weeks. Thus there only remains the assumption that in the fluorescing uranium salts part of the Becquerel radiation is converted into light energy, and this conversion is greater the more strongly the salts fluoresce.—*Berichte*, 1906, xxxix., p. 200.

ANALYSIS OF ALLOYS OF COPPER.*

By JOHN M. WILSON,
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(Concluded from p. 85).

Phosphor Bronze.

WEIGH 1 grm. into a No. 1 beaker, dissolve in 5 c.c. concentrated HNO_3 , warm on a steam plate till free from nitrous fumes, and add 25 c.c. water. Warm until precipitated SnO_2 is white, filter, wash with HNO_3 (1 to 10), and electrolyse for copper and lead.

WEIGH 5 grms. into a No. 3 beaker, add 25 c.c. concentrated HNO_3 , and heat till free from nitrous fumes. Add 75 to 100 c.c. water, digest till insoluble matter is white, filter through a double filter, and wash copper salts out with HNO_3 (1 to 10). Put filter and precipitate in an Erlenmeyer flask, add 25 c.c. $(\text{NH}_4)_2\text{S}$, digest two to three hours in the cold, shaking occasionally, and heat on steam-plate for one hour. Filter, wash out all alkaline sulphides,

* The Chemical Engineer, ii., No. 3.

add 20 to 25 c.c. magnesia mixture, and shake occasionally for twenty minutes. Add one-sixth volume concentrated NH_4OH , shake well, allow to stand over-night, and filter into a 500 c.c. graduated flask. Wash out all $(\text{NH}_4)_2\text{S}$, dissolve, precipitate in HCl , and re-precipitate with NH_4OH . Add 100 c.c. $(\text{NH}_4)_2\text{S}$, digest on steam-table for one hour, add $\frac{1}{2}$ volume of NH_4OH , allow to stand several hours, filter into the same flask, wash thoroughly, and take up in HCl . Evaporate to dryness, moisten with HCl , take up in water, add H_2SO_4 , boil, saturate with H_2S to get rid of any possible As , filter, and evaporate off H_2S . Cool thoroughly, add NH_4OH till neutral, shake vigorously, keeping the liquid cold, and add NH_4OH , drop by drop, until the precipitate forms. Set aside fifteen minutes, shaking occasionally, and add one-sixth its volume of NH_4OH . Shake vigorously, allow to stand at least four hours, filter on a weighed Gooch felt, wash with NH_4OH (1 to 3) containing NH_4NO_3 , dry at 100°C ., ignite, and weigh as $\text{Mg}_2\text{P}_2\text{O}_7$. Calculate to P .

Make the sulphide filtrate up to mark and mix thoroughly, take 100 c.c. (equivalent to 1 grm.), add 1 to 2 grms. KClO_3 , and then 25 c.c. concentrated HCl . Digest on the steam-plate, adding KClO_3 if required, until the separated sulphur is a clear yellow colour, filter through glass-wool, and wash with hot water. Transfer to a No. 3 beaker (volume 200 to 250 c.c.), add a slight excess of NH_4OH , and then 10 grms. $\text{H}_2\text{C}_2\text{O}_4$ + 10 grms. $(\text{NH}_4)_2\text{C}_2\text{O}_4$. Warm till a clear solution is obtained, and electrolyse over-night with a current of 5 c.c. electrolytic gas per minute. Wash, dry, and weigh the electrode as for copper. The weight represents the tin. Iron is to be determined as in manganese bronze.

Babbitt Metal and White Metals.

For the analysis of these metals one of two methods is used, according to the quantity of lead present.

Little Lead Present.

Five grms. of cuttings are weighed into a 5-inch dish, concentrated, 50 c.c. HNO_3 are added, and when violent action ceases, the solution is evaporated to dryness on the steam-table. The dry powder is moistened with 20 per cent NaOH , 10 to 20 c.c. being required, 40 c.c. of Na_2S is added, and after boiling one hour 80 c.c. water is also added. The residue is allowed to settle, the clear liquid is decanted as much as possible into an Erlenmeyer flask, and the digestion repeated twice, using 30 c.c. and 20 c.c. Na_2S respectively. The solution is then filtered through a double filter, or preferably a pulp filter as recommended by Shimer, washing out all alkaline sulphides with cold water, keeping the filter completely filled with wash all the time. When washed the filter is allowed to drain. Dead roast the precipitate in porcelain, dissolve in HNO_3 , and electrolyse the copper and lead.

Make the residual liquid and washings in the Erlenmeyer flask slightly alkaline, warm, and precipitate with H_2S . Cork flask and allow to stand over-night. Filter through paper, wash with water containing $(\text{NH}_4)_2\text{S}$, and dissolve soluble sulphides in a mixture of 1 volume HCl + and 4 of H_2S water. Wash with cold water, ignite, and weigh precipitate. Test to ascertain whether it is copper or nickel.

Evaporate the solution of soluble sulphides to dryness, moisten with HCl , add a few drops HNO_3 , and again evaporate to dryness. Take up with HCl and water, precipitate the iron three times with NH_4OH , and dissolve the precipitate in H_2SO_4 . Reduce, and titrate the iron.

Saturate the cold united filtrates with H_2S , allow to stand over-night, filter through a double filter. Wash with cold water, ignite in porcelain, and weigh as ZnO . Calculate to zinc.

Make alkaline sulphide solutions up to volume, mix thoroughly, and take out four portions:—Two of 50 c.c. each (equivalent to $\frac{1}{2}$ grm.), and one of 25 c.c. (equivalent to $\frac{1}{4}$ grm.), and one of 20 c.c. (equivalent to $\frac{1}{5}$ grm.).

The 20 and 25 c.c. portions are to be oxidised with

$\text{HCl} + \text{KClO}_3$ and electrolysed for tin exactly as described under phosphor bronze.

The antimony is determined either (a) by Clarke's method modified, or (b) by Lord's method modified, or both.

(a). *Clark's Method*.—Dilute the 50 c.c. portion to 400 c.c., add 30 grms. $\text{C}_2\text{H}_4\text{O}_2$, heat on steam-table until all dissolves except a few flocks of Sb_2S_3 , boil ten minutes, and pass a rapid current of H_2S for twenty minutes. Allow to stand in a warm place until the odour of H_2S is nearly gone. Filter through paper, wash with hot water, test filtrate for any unprecipitated antimony. Wash precipitate back into beaker in which it was made, place beaker under funnel, dissolve adhering Sb_2S_3 from paper with a few drops NaOH (1 to 20), and reserve the filter (do not wash). Add 10 to 15 c.c. NaOH (1 to 10) to contents of beaker, warm till all Sb_2S_3 is in solution, filter through the same filter, wash well with hot water, and add 20 grms. $\text{C}_2\text{H}_4\text{O}_2$. Repeat the precipitation. Filter and repeat the solution and precipitation, filter finally on asbestos felt in a small carbon tube, wash with hot H_2O , then with alcohol, then with CS_2 , then with alcohol, and finally with ether. Dry at 100°C ., and introduce carbon tube into a large straight tube. Close end of this tube with a stopper carrying a bent delivery tube, and pass a current of CO_2 through it. Heat to 300°C ., when S is given off and the precipitate turns black. When no more S is given off cool in the streams of CO_2 and weigh Sb_2S_3 . Calculate to antimony.

(b). *Lord's Method*.—Dilute the 50 c.c. portion to 400 c.c. Add a slight excess of HCl , allow to settle, filter through a double filter. (A Shimer pulp filter would answer better here for convenience in subsequent handling). Wash with water containing $(\text{NH}_4)_2\text{H}_2\text{C}_2\text{O}_4$. Remove precipitate as far as possible with a spatula to the beaker in which it was made, dissolve adhering precipitate with a few drops NaOH (1 to 10), and wash once with water, keeping the volume down. Add enough NaOH (1 to 10) to dissolve precipitate, filter through the same filter into a No. 2 beaker, and wash the filter thoroughly with water, keeping volume below 80 c.c. If more than that volume evaporate to 80 c.c., add one stick solid NaOH (about 10 to 15 grms.), and when dissolved, cool solution. Add 3 c.c. liquid bromine, allow to stand at laboratory temperature over-night, and in the morning place a drop of the solution on the cover. Add a drop of HCl , and if bromine fumes are given off proceed as below; if not add more bromine and digest two to three hours, test again.

When sufficient bromine is present warm on the steam-table one hour and then boil ten minutes. Cool, add one-third its volume of alcohol (95 per cent), stir well, and allow to stand over-night.

Filter through paper, and wash with a mixture of alcohol, water, and Na_2CO_3 . Dissolve precipitate in $\text{HCl} + \text{C}_2\text{H}_6\text{O}_6$, dilute to 300 c.c., add 10 grms. $\text{C}_2\text{H}_2\text{O}_4$, boil and saturate with H_2S by passing a rapid current for twenty minutes. Filter through paper, dissolve in NaOH , re-precipitate the Sb_2S_3 with $\text{C}_2\text{H}_2\text{O}_4 + \text{H}_2\text{S}$, filter through asbestos felt in carbon filtering tube, wash with hot water, alcohol, CS_2 , alcohol and ether, dry at 100°C ., heat to 300°C in current of CO_2 in a larger tube, cool in CO_2 , and weigh Sb_2S_3 as directed before. Calculate to antimony.

Both of the above methods give accurate results after the conditions have been mastered, and the results obtained by them check beautifully.

II. When much lead is present I use a method proposed by Mr. G. W. Thompson in "Stillman's Engineering Chemistry."

Weigh 1 or more grms. of cuttings into a beaker, add 80 c.c. per grm. taken of the solvent for white metals given above, evaporate to a syrup, cool rapidly with constant stirring, and add 100 c.c. alcohol (95 per cent), stirring the while. Allow to stand twenty minutes, filter, and wash with a mixture of $\text{HCl} + \text{alcohol}$. Dissolve the precipitate in hot water and $(\text{NH}_4)_2\text{H}_2\text{C}_2\text{O}_4$, warm, boil the solution, and precipitate and weigh the lead as PbCrO_4 .

Evaporate filtrate to dryness, moisten with 10 to 20 c.c. NaOH (10 per cent), add 20 to 40 c.c. H_2O_2 , warm on steam-table for twenty minutes, and add 10 to 20 grms. $C_2H_2O_4$ and 300 c.c. water. Boil the solution, and pass a rapid stream of H_2S through it for twenty minutes. Filter and wash with hot water. Wash the precipitate back into the beaker in which it was made, run 20 c.c. NaOH (1 to 20) through the filter, and wash the latter once with water. Reserve the filter, and add more NaOH to the filtrate and digest on the steam-table until the precipitate has lost all red colour and is quite black. Filter through the same filter, washing thoroughly with water, and dilute the filtrate to 300 c.c. Add 10 grms. $C_2H_2O_4$, heat till all is dissolved except a few flocks of Sb_2S_3 , boil, saturate the solution with H_2S for twenty minutes, and filter on asbestos in a carbon filtering tube. Wash, ignite in CO_2 as above, weigh, and calculate to antimony. Unite the oxalate filtrates, make up to volume, take a suitable portion, and electrolyse for tin.

Burn the black precipitate in porcelain, react until free from sulphur, dissolve in HNO_3 , electrolyse for copper and lead, and in the residual liquid determine iron, zinc, &c.

COMPARISON OF A
WET AND CRUCIBLE-FIRE METHODS FOR
THE ASSAY OF GOLD TELLURIDE ORES,
WITH NOTES ON THE
ERRORS OCCURRING IN THE OPERATIONS
OF FIRE ASSAY AND PARTING.*

By W. F. HILLEBRAND and E. T. ALLEN.

INTRODUCTION.

THE scorification assay for telluride ores has long been believed, and apparently proved, to give low results by reason of volatilisation, and it is now seldom used for ores of that class. The assay by crucible is supposed to be far more reliable, and Furman maintains that it is entirely so when properly carried out ("Manual of Practical Assaying," 1899, p. 399). He gives a very few comparative determinations by fire and wet assay in proof of his assertion, but the tests are too few in number and the agreement is not so close as might be desired. A more critical investigation of this problem seemed to promise results of some value to those engaged in mining and smelting the telluride ores which are so important in certain parts of our own and other countries, and the data and conclusions set forth in the following pages are the outcome of experiments on telluride ores from Cripple Creek, Colorado. From them it will be seen that there is no occasion to question the substantial accuracy of the crucible method, as tested by careful determinations in the wet way. Incidentally the question of losses of gold in assaying, particularly during cupellation and parting, has been looked into, for concerning these points the most contradictory statements are to be found in the literature. Only after most of the work upon the ores had been completed was this fully impressed upon us and the extent of some of these losses duly appreciated. Had this appreciation come earlier we might have been able to offer still more satisfactory results for our assays.

PART I. ASSAY OF CRIPPLE CREEK TELLURIDE ORES.

Nature of the Ores Assayed.

Efforts were made to procure two ores of somewhat widely differing gold content and, with a view to the preparation of a homogeneous sample, containing as little free gold as possible. As received, through the kindness of Mr. W. M. Bainbridge, of the El Paso Consolidated Gold Mining Company, and of Dr. Waldemar Lindgren,

of the Geological Survey, the samples were marked 6 and 12 ounces per ton respectively, but they were actually much richer and of nearly equal value, approximately 15 and 19 ounces to the ton. At least 10 pounds of each was available. The ores were from a narrow fissure in granite, and were themselves practically granite, carrying pyrite in some quantity, the higher grade sample *b* more than *a*; some calcite; also traces of copper and molybdenum, as shown by determinations on 100 grms. Other constituents were not looked for. The tellurium also was quantitatively determined, in 50 grm. portions, in order to judge of the composition of the telluride mineral. As given below, silver is, no doubt, a trifle low, because of the impossibility of making exact allowance for loss in assaying.

TABLE I.—Amounts of certain Constituents of the Ores.

	a (per cent).	b (per cent).
Te	0.0742	0.092
Au	0.0506	0.060
Ag	0.0075	0.0103
Cu	0.0059	0.0070
Mo	0.0015	0.0018

Assuming none of the gold and silver to be free, but all in combination with tellurium, the following percentage compositions for the telluride have been calculated from the foregoing table:—

Calculated Composition of the Tellurides.

	a.		b.	
	Per cent.	Atomic ratio.	Per cent.	Atomic ratio.
Te ..	56.09	1.78	56.68	1.80
Au ..	38.24	1.00	36.97	1.00
Ag ..	5.67		6.35	
	100.00		100.00	

If the tellurium has been correctly determined, the above results indicate the probable presence of free gold, for from what we know of the occurrence of gold-silver tellurides at Cripple Creek they probably all conform to the general formula MTe_2 . That the telluride here present must then be largely if not wholly sylvanite, assuming absence of other silver minerals, is indicated by the amount of silver shown by the analyses, which is considerably in excess of that in even the richest known calaverite. It is not impossible that both calaverite and sylvanite as well as free gold are present. From the ratio of gold to silver in a second pair of samples, a_1 , and b_1 , which, as mentioned later, were taken from a lower position in the original sample bags, so that a considerable mechanical sorting had ensued since the bags were filled, the suspicion may be justified that the silver is in part a constituent of some mineral other than a telluride. For if the assays are correct there is a diminution in the silver as the gold increases, as shown by the following comparison:—

	a (ozs.).	a_1 (ozs.).	b (ozs.).	b_1 (ozs.).
Ag ..	2.19	2.07	3.00	2.87
Au ..	14.53	15.60	17.75	19.63

That not all the silver may thus be assigned elsewhere is, however, likely from the fact that the ratio of tellurium to gold alone is much in excess of 2 to 1. But too much weight must not be attached to the above values for silver, and therefore speculations of this sort are of little importance.

Preparation of the Samples.

Considerable portions from the tops of the bags containing the coarsely crushed samples received were ground fine enough to pass a sieve of 100 meshes to the linear inch and mixed for what was considered a sufficient time to insure homogeneity. The amounts thus prepared proved inadequate for the completion of the work, so other portions were ground fine enough to pass a sieve of 150 meshes and mixed more intimately even than in the first instance; this

* United States Geological Survey. *Bulletin* No. 253. (Series E, Chemistry and Physics, 44).

being done because the first results on sample *b* were somewhat less satisfactory and less accordant than those on *a*, a condition which it was hoped finer grinding would remedy. These second samples were found to be markedly richer in gold than the first, due doubtless to their having been taken from a greater depth in the original bags. They represented practically new ores, and, to simplify references, will be designated *a*₁ and *b*₁.

Combination Wet and Fire Assay.

Throughout the literature it is prescribed that for wet assay of gold ores they should be subjected to a preliminary roasting for the removal of sulphur chiefly. Since, however, it is pretty certain that there is loss of gold by volatilisation in the roasting of telluride ores, it seemed imperative to disregard these directions and to treat chemically the unroasted ore. No attempts were made to test different methods of attack and subsequent treatment because of lack of time. The method employed in all cases was the following:—

Fifty-grm. portions in duplicate were mixed with water in large porcelain basins, and strong nitric acid was added by degrees with constant stirring till action had nearly ceased. Then bromine water was added in excess to oxidise the gold and sulphur and precipitate the silver. No artificial heat was employed. The following morning the insoluble matter was collected on filters and washed ten times with water containing bromine and some hydrochloric acid, then two or three times with pure water.

It was found impossible to extract the whole of the gold by wet treatment. The residues always yielded by fire assay small amounts of gold, usually under 0.1 mg. when properly washed, besides nearly all the silver.

The combined filtrates were evaporated in the original porcelain basins to near dryness, then treated with hydrochloric acid and covered for a time. When action ceased the covers were removed and evaporation continued, hydrochloric acid being added two or three times more at proper intervals. When the residues were now digested with dilute hydrochloric acid and a little bromine water, to insure solution of the gold, there remained undissolved considerable calcium sulphate, which was collected on a suitable-sized filter and the filtrate received in a beaker. The first calcium sulphate residues thus obtained were found by fire assay to be free from gold, but all those obtained in subsequent tests were added to and assayed with the main residues.

The filtrates contained all the iron in an oxidised form and a slight excess of bromine. Without attempting to reduce the ferric iron to ferrous by sulphurous acid, whereby tellurium would have been in part precipitated as well as the gold, a large excess of filtered ferrous sulphate solution was added, which induced complete precipitation of the gold alone, without any tellurium. The completeness of precipitation was proved by the failure to detect any gold in the subsequently precipitated tellurium.

After twenty-four hours the precipitated gold was collected on a 9 c.m. paper filter of 0.00005 grm. ash content, carefully washed with cold water, then with warm dilute hydrochloric acid to extract iron from the paper, and finally with hot water. Paper and contents were burned in a clean porcelain crucible, and the contents most carefully transferred to the pan of the assay balance and weighed as nearly as might be to hundredths of a m. grm. As a check the gold was then generally cupelled with 3 or 4 grms. of pure lead in order to eliminate the error introduced by paper ash and any possible failure to wash the filter thoroughly. A companion cupellation with a similar amount of proof gold afforded the correction for gold loss during cupellation. A number of the beads thus obtained from the ore were collectively tested most carefully for silver, with negative results.

It was said above that it was not possible to extract quite all of the gold from the ore, and that the residues insoluble in acid were treated by fire assay in order to recover the

amount retained. The retention was probably owing to particles of telluride inclosed in gangue matter, or of gold in sulphur which had been separated but not fully oxidised by the treatment with nitric acid and bromine. These residues were assayed in crucibles with the following charge: Litharge, 2 A. T.; soda, 1 A. T.; borax, 10 grms.; argol, 2 grms.; salt, cover. The lead button thus obtained weighed about 20 grms. and yielded on cupellation nearly all the silver of the ore and a small amount of gold, which latter was obtained pure by direct parting. It was not deemed necessary to examine slags or cupels for their possible gold contents, owing to the minute amount in question, nor was any attempt made to determine by the wet way the exact amount of silver that the ore held.

TABLE II.—*Combination Wet and Fire Assays for Gold in Cripple Creek Ores.*

(Fifty grms. in each case).

Residues. M.grm.	By FeSO ₄ . M.grms.	Total.		After cupellation.	
		M.grms.	Ozs. per ton.	M.grms.	Ozs. per ton.
Sample a.					
0.08	25.29	25.37	14.80	—	—
0.04	25.22	25.26	14.73	25.22	14.71
0.08	25.73	25.85	15.08	25.42(a)	14.82
0.06	25.63	25.69	14.98	25.30(a)	14.76
Sample a ₁ .					
0.12	26.48	26.60	15.52	26.51	15.46
0.16	26.32	26.48	15.45	26.45	15.43
0.05	26.69	26.74	15.60	26.59	15.51
0.12	26.89	27.01	15.76	26.73	15.59
Sample b.					
0.13	—	—	—	—	—
0.12	29.56	29.68	17.32	29.45	17.18
0.07	30.84	30.91	18.03	30.57	17.83
0.12	30.50	30.62	17.86	30.17	17.60
Sample b ₁ .					
0.98	32.54	33.52	19.55	33.35	19.45
0.37	33.13	33.50	19.54	33.32	19.44
0.10	33.10	33.20	19.58(b)	33.07	19.49(b)
0.11	33.85	33.96		33.76	

(a) These beads contained a very little silver, which was lost in process of parting.

(b) The assays were made in pairs, and by accident a part of the washings of one assay was evaporated in the wrong dish. The assays were run through separately, but only the mean is taken for final comparison.

There is seen to be a very fair agreement in each of the several series except *b*, an exception which was apparent also in the fire assay tests of the ore. It was largely for this reason that fresh samples of each ore were prepared (*a*₁ and *b*₁), as before mentioned.

(To be continued).

Educational Post Cards.—The Country Press, of 19, Ball Street, Kensington, W., are issuing a novel series of Educational Post Cards, the first example of which (Natural History Department) is a picture presentment, on seven cards, for the price of sixpence, of the whole of the British Ferns (42 species, nature prints) from the illustrative plates of Mr. Francis George Heath's work, "The Fern Paradise." These are to be followed by other representations on post cards of natural history subjects and others likely to have an educational value.

CONDENSATION COMPOUNDS OF CAMPHOR-
OXALIC ACID AND AMINES.*SEVENTH COMMUNICATION ON CAMPHOROXALIC
DERIVATIVES.By J. BISHOP TINGLE, Ph.D., F.C.S.,
and
WILLIAM EDWIN HOFFMAN, Jun., Ph.D.

(Continued from p. 88).

ACTION OF AMINES ON CAMPHOROXALIC ACID (continued).

XI. 1,2,4-Nitrotoluidine.

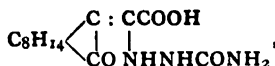
When nitrotoluidine ($\text{CH}_3 = 1$, $\text{NH}_2 = 2$, $\text{NO}_2 = 4$) is heated at 150° with camphoroxalic acid in alcoholic solution under pressure for several hours, *nitrotolylcamphorformeneamine*,—



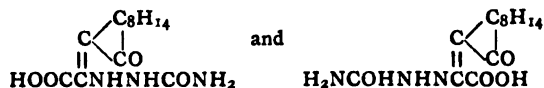
is obtained. It crystallises readily from alcohol in minute bright yellow needles which melt at 192° . It was not possible to isolate the corresponding carboxylic acid or salt, although a number of experiments under varying conditions were made with this object.

XII. Semicarbazine.

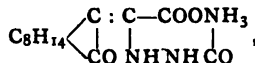
When semicarbazine and camphoroxalic acid react in the presence of acetic acid, a compound is formed with the composition of *semicarbazine camphorformeneaminecarboxylic acid*,—



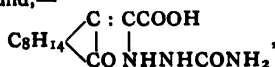
It crystallises from alcohol in stout colourless prisms melting at 200° . When dissolved in boiling glacial acetic acid, crystals were deposited in the form of clusters of fine needles which melt at $209\text{--}210^\circ$. If the compound obtained from glacial acetic acid is dissolved in sodium carbonate and re-precipitated by acid, a gelatinous substance is formed, which crystallises from acetone in slender colourless needles melting at the same temperature as before, viz., $209\text{--}210^\circ$. These two substances have the same empirical formula and maintain their distinctive melting-points even after heating for some time at 110° . They dissolve readily in sodium-hydrogen carbonate, and are re-precipitated by acids in the form of a jelly. With alcoholic ferric chloride, no colour reaction is produced in either case. All these facts lead to one of two conclusions: either the compounds are stereoisomers of the maleic-fumaric type,—



or there is the formation of an inner salt,—

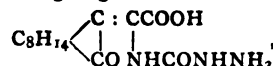


which is changed by the glacial acetic acid into the open-chain compound,—



In the case of these semicarbazine derivatives we have represented the condensation as having taken place between the hydrazine nucleus and the camphoroxalic acid. Although this is in accordance with precedent, it may well

be that in one or both compounds it is the carbamide group which has reacted, giving—



and its stereoisomer. Further work will be necessary to decide between these various possibilities; the subject is being more fully investigated in the laboratory of the Johns Hopkins University.

XIII. Orthophenylenediamine.

When orthophenylenediamine and camphoroxalic acid are allowed to react in alcoholic solution at the ordinary temperature, there is formed the camphoquinoxaline compound, which was prepared by J. Bishop Tingle by the action of sodium camphoroxalate, and also of ethylcamphoroxalate, on orthophenylenediamine. It crystallises from alcohol in bright yellow needles which melt at 246° . The formation of this camphoquinoxaline by the interaction of sodium camphoroxalate, ethylcamphoroxalate, or camphoroxalic acid respectively, is somewhat remarkable, as it shows the tendency of such a phenylenediamine derivative, in which the amino-groups are in the ortho-position, to form a five-membered ring compound even under conditions where we should not expect such an action to take place.

Paraphenylenediamine was treated with camphoroxalic acid under the same conditions as were used for the ortho-compound, and also under others, in the hope that it might react and throw some light on this question of ring formation, but no definite product could be obtained.

(To be continued).

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, February 15th, 1906.

Prof. R. MELDOLA, F.R.S., President; in the Chair.

MESSRS. H. B. Hartley and H. L. Tidy were formally admitted Fellows of the Society.

Certificates were read for the first time in favour of Messrs. Richard Henry Beckett, B.Sc., Inval, Haslemere, Surrey; Herbert Reginald Cooper, Redington, Northwood, R.S.O.; Charles Davidson, 37, Herriot Street, Pollokshields, Glasgow; Edward Gardner, 70, Parliament Hill Mansions, Highgate Road, N.W.; Richard Godfrey Hamilton Garvey, 25, Park Mansions, Battersea; William Tabor Lattey, Corpus Christi College, Cambridge; Hamilton McCombie, M.A., B.Sc., Ph.D., The University, Birmingham; Thomas Jenkins Murray, Ph.D., The University, Birmingham; Edgar Gall Oliver, M.A., Chigwell School, Essex; Lawrence George Richardson, 14, Ashgrove, Horton, Bradford; David Somerville, B.A., M.D., 31, Manor House, Marylebone; Foster Sproxtton, B.Sc., Uplands, Alexandra Park Road, Wood Green, N.; Harold Augustine Tempamy, B.Sc., St. Johns, Antigua, B.W.I.

It was announced that the following changes in the Officers and Council were proposed by the Council:—

As Vice-Presidents—Prof. W. H. Perkin, jun., F.R.S., and Dr. Rudolph Messel, vice Prof. W. R. Dunstan, F.R.S., and Mr. D. Howard.

As Ordinary Members of Council—Prof. W. Gowland, Dr. H. A. D. Jowett, Dr. F. E. Matthews, and Prof. A. G. Perkin, F.R.S., vice Prof. A. E. Dixon, Prof. J. J. Dobbie, F.R.S., Dr. E. J. Mills, F.R.S., and Prof. J. M. Thomson, F.R.S.

Mr. E. Grant Hooper, Dr. H. F. Morley, and Dr. H. R. Le Sueur were elected Auditors to audit the Society's accounts.

* From the *American Chemical Journal*, xxxiv., No. 3.

A ballot for the election of Fellows was held, and the following were subsequently declared duly elected:—George Ernest Banner; Arthur Ernest Barker, B.A., B.Sc.; William Edward Bell; Charles Frederick Polwhele Blatchley, B.A.; Thos. Going Stoney Bogue; Arthur Forbes Braid; Richard Victor Briggs; William Caldwell, M.A.; Reginald William Lane Clarke; Wm. Boulton Conyngham; Stephen Lewis Courtauld, B.A.; Thomas Harold Durrans; Frederick Watkins Evans; Hans Eduard Fierz, Ph.D.; Walter Hamis Glover, Ph.D.; Harry Sands Grindley, D.Sc.; William Prince Hayworth; Henry Carlyle Irving, B.A.; Llewellyn Thomas Jones, B.Sc.; William Henry Matthews Jones; Robert Le Rossignol, B.Sc.; John McDowall; William Mace; George Frederick Wesley Martin; Samuel Parfitt; John Parkin, M.A.; William Hughes Perkins, B.Sc.; John Edmund Pitman, B.Sc.; Harold Rogerson, M.Sc.; Philip Foale Rowsell; John Kirby Shrimpton; Frank Smith, B.Sc.; George Stanley Talbot; Percy Charles Thornton; Arthur William Thorp; Ronald William Tonkin; Franklin Wilfred Walker; Richard Alfred Warren; Hardolph Wastenays; Henry Edgar Watt, M.Sc.; Elkan Wechsler, Ph.D.; Harold Joseph Wheaton; Herbert William Mills Willett; Richard Willstaetter, Ph.D.; Ernest Edwin Wolfe; John William Yates, B.Sc.

Of the following papers, those marked * were read:—

*35. "Cuprous Formate." By ANDREA ANGEL.

The compound described as cuprous formate by Joannis (*Abstr.*, 1904, i., 644), having the formula $(\text{HCO}_2)_2\text{Cu}_2\cdot 4\text{NH}_3\cdot \frac{1}{2}\text{H}_2\text{O}$, should be named ammonio-cuprous formate, since it is quite different in properties from the substance prepared by dissolving cuprous oxide in an aqueous ammoniacal solution of ammonium formate under petroleum, diluting the solution with alcohol, and acidifying with formic acid, when crystals are deposited which are collected in an atmosphere of hydrogen and washed with ethyl formate. *Cuprous Formate*, $(\text{HCO}_2)_2\text{Cu}_2$, consists of colourless crystals which are very easily decomposed; water immediately hydrolyses the substance to cuprous oxide and formic acid, sodium carbonate solution decomposes it with effervescence, ammonia solution decomposes it with a slight hissing noise, and dilute sulphuric acid at once produces a precipitate of metallic copper. Dilute formic acid acts similarly, although more slowly. Moist air rapidly hydrolyses it, turning it orange-red, but in a desiccator over sulphuric acid it may be preserved apparently for an indefinite time. Owing to the ease with which the substance is decomposed, the preparation requires special precautions in regard to the strength of the solutions employed. The method of preparation may be employed, with modifications, for the production of other cuprous salts.

DISCUSSION.

Mr. A. VERNON HARCOURT said that Mr. Angel's research arose out of some experiments on the gradual heating of cupric acetate in a vacuum, an account of which had already appeared in the *Transactions*.

Both cupric acetate and cupric formate when strongly heated in a glass vessel deposit a mirror of copper on the glass, showing that a gas containing copper has been formed. The temperature of decomposition of cuprous acetate is very near its temperature of volatilisation, and neither it nor cuprous formate can be obtained, except in very small proportion, by sublimation from the cupric salt.

*36. "The Solubility of Triphenylmethane in Organic Liquids, with which it Forms Crystalline Compounds." By HAROLD HARTLEY and NOEL GARROD THOMAS.

Experiments were made on the crystallisation of triphenylmethane from a large number of organic liquids in order to see in what cases a crystalline compound is formed with the solvent. From benzene, thiophen, pyrrole, and aniline solutions, it crystallises under certain conditions with one molecule of the solvent, and a microscopic study of crystals of the four compounds has shown that they all

belong to the rhombohedral system, forming an isomorphous series.

The solubility of triphenylmethane in each of the four liquids and also in pyridine has been determined by a modification of the method devised by Kuriloff. Weighed quantities of solvent and solute are sealed in small glass tubes, and these are placed in a water-bath. By varying the temperature of the bath, two temperatures can be determined, at the lower of which the last crystal left undissolved is growing, and at the upper the same crystal is dissolving. These temperatures are usually 0.4° to 0.5° apart, and their mean has been taken as the temperature of saturation.

Experiments were also made on the spontaneous crystallisation of supersaturated solutions of triphenylmethane in each of the foregoing solvents by cooling solutions in sealed tubes after they had been heated sufficiently to destroy all crystalline nuclei. When the tubes were shaken during the cooling, crystallisation took place at definite temperatures. The results confirm the existence of a metastable region in which supersaturated solutions cannot crystallise spontaneously. The limit of this region where the solutions pass into the labile state and crystallise can be represented by a curve, for which Miers and Isaac have suggested the name "supersolubility curve." It runs approximately parallel to the solubility curve at a distance from it depending on the respective solvent and solute.

*37. "The Spontaneous Crystallisation of Supersaturated Solutions." By HAROLD HARTLEY.

The author discussed the different views of Ostwald and de Coppet on this subject in view of the experiments described in the preceding communication, and those of Miers and Isaac (*Proc.*, 1906, xxii., 9). It was shown how the difference between metastable and labile solutions might be explained from the kinetic standpoint as a result of the increased solubility of the small crystals which must be first formed in a spontaneous crystallisation.

DISCUSSION.

Prof. MIERS welcomed these investigations as new examples of the importance of the supersolubility curve, and called attention to the fact that the authors could not have established their results without a practical knowledge of crystallography.

Sir WILLIAM RAMSAY pointed out the analogy between the behaviour of crystallisable solution in the metastable and stable states with that of a super-heated liquid, and suggested that Mr. Hartley might derive some help by a study of such behaviour.

Mr. HARTLEY, in reply, said that small crystals besides being abnormally soluble must also have a lower melting-point than larger individuals, and that this fact would explain the usual occurrence of superfusion. Mr. P. W. Robertson and he were just starting to investigate the conditions under which a number of pure organic substances crystallise spontaneously, and the former had suggested that the variation of melting-point with the size of a crystal might explain some of the irregularities which had been observed in cryoscopic work with such solvents as thymol. Supersaturated solutions did not crystallise immediately on reaching the labile state unless they were vigorously shaken; thus the pull on the solution, which was suggested by Sir William Ramsay's analogy of the continuous isothermal as necessary to start crystallisation, was actually realised in the continuous production of fresh liquid surfaces.

*38. "Preparation and Properties of some New Tropeines." By HOOPER ALBERT DICKINSON JOWETT and ARCHIE CECIL OSBORN HANN.

The object of this investigation was primarily to determine whether any difference in physiological action could be observed between tropeines containing a lactone grouping and their corresponding hydroxy-acid, similar to that recorded in the case of pilocarpine and pilocarpic acid (Marshall, *Journ. Physiol.*, 1904, xxxi., 153). For this purpose, the tropeines of methylparaconic, terebic, and phthalidecarboxylic acids were prepared, when it was

found that both terebyl- and phthalidecarboxyl-tropeines, which produce an atropine-like effect on the heart, lose this action after a molecular proportion of alkali has been added to the base, thus showing in aqueous and alkaline solution a difference in action analogous to that observed in the case of pilocarpine.

In addition to the tropeines already mentioned, the glycolyl- and protocatechyl-derivatives were prepared and the opportunity taken to test Ladenburg's generalisation. According to this chemist, a tropeine, in order to possess mydriatic action, must contain a benzene nucleus and a fatty hydroxyl attached to the same carbon atom as that bearing the carboxyl group. It was found that this did not strictly hold, as terebyltropeine possessed a distinct mydriatic action. It would appear, however, that the conditions most favourable for the development of the mydriatic action in a tropeine are those stated by Ladenburg, namely, that the acyl group should contain a benzene nucleus and an aliphatic hydroxyl in the side-chain containing the carboxyl group.

*39. "Studies in Asymmetric Synthesis. IV. The Application of Grignard's Reaction for Asymmetric Syntheses." By ALEXANDER MCKENZIE.

The author has studied the action of magnesium propyl iodide, magnesium isobutyl iodide, and magnesium α -naphthyl bromide respectively on *l*-menthyl benzoylformate, and effected in each case an asymmetric synthesis of a substituted *l*-glycollic acid. The influence of the *l*-bornyl as contrasted with that of the *l*-menthyl grouping was examined by acting on *l*-bornyl benzoylformate with various magnesium alkyl halides; the effect of the *l*-bornyl grouping was to diminish the laevorotation of the mixture of substituted glycollic acids obtained; thus, whilst the mixture of *d*- and *l*-atrolactic acids obtained from *l*-menthyl benzoylformate and magnesium methyl iodide had $[\alpha]_D -9.5^\circ$ in ethyl-alcoholic solution, the mixture from *l*-bornyl benzoylformate under similar conditions gave $[\alpha]_D -1.9^\circ$.

A mixture of *d*- and *l*-phenylisobutylglycollic acids containing an excess of the *d*-acid was produced from *l*-bornyl benzoylformate and magnesium isobutyl iodide, whereas the mixture was laevorotatory when *l*-menthyl benzoylformate was used. Similarly, a dextrorotatory mixture of acids was produced by the action of *l*-bornyl benzoylformate on magnesium α -naphthyl bromide.

The asymmetric synthesis of *d*-atrolactic acid was produced by the action of magnesium phenyl bromide on *l*-menthyl pyruvate.

When *l*-menthyl acetoacetate, *l*-menthyl ethylacetate, or *l*-menthyl diethylacetate is submitted to the Grignard reaction, no asymmetric synthesis occurs, the first of these substances reacting in accordance with its enolic structure. A slight asymmetric synthesis was, however, detected when *l*-menthyl laevulate was used.

40. "o-Cyanobenzenesulphonic Acid and its Derivatives." By A. JAMIESON WALKER and ELIZABETH SMITH.

Reference was made to the work of Jesurun (*Ber.*, 1893, xxvi., 2288) on *o*-cyanobenzenesulphonic chloride, and to that of Remsen and others (*Am. Chem. Journ.*, 1895, xvii., 309 and 347; *Ibid.*, 1896, xviii., 794 and 819), and List and Stein (*Ber.*, 1898, xxxi., 1648) on ammonium *o*-cyanobenzenesulphonate.

The authors then described a modification of Jesurun's method for the preparation of *o*-cyanobenzenesulphonic chloride and the isolation from the mother-liquor of *o*-cyanobenzenesulphonic acid, $\text{CN}\cdot\text{C}_6\text{H}_4\cdot\text{SO}_3\text{H}$, which crystallises from water in white needles melting at $279-279.5^\circ$. Its silver salt has been prepared, and the action of nitric acid and bromine on the acid investigated.

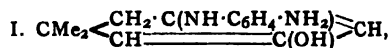
Reduction of the chloride with zinc dust yields *o*-cyanobenzenesulphinic acid, $\text{CN}\cdot\text{C}_6\text{H}_4\cdot\text{SO}_2\text{H}$, which crystallises in small white needles melting at $226.5-228^\circ$. With bromine, this acid yields two compounds melting at $156-156.5^\circ$ and $172.5-173^\circ$ respectively, but containing no bromine. Their constitution is still being investigated. Nitrous acid converts the sulphonic acid into a yellow

solid, probably *o*-cyanodibenzsulphohydroxamic acid, $\text{N}(\text{C}_6\text{H}_4(\text{CN})\cdot\text{SO}_2)_2\cdot\text{OH}$.

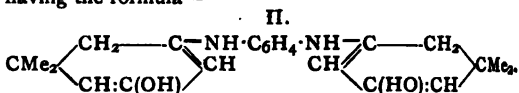
Heating with dilute caustic soda and subsequent acidification transform the chloride into a substance (m. p. $221.5-223^\circ$) with the sweet taste of "saccharin," and not into the sulphonic acid.

41. "The Condensation of Dimethyldihydroresorcin and of Chloroketodimethyltetrahydrobenzene with Primary Amines. Part II. Diamines; *m*- and *p*-Phenylenediamines." By PAUL HAAS.

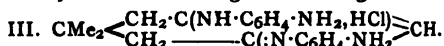
Dimethyldihydroresorcin condenses, in alcoholic solution, with one molecular proportion of a diamine, giving an 80 per cent yield of the compound—



together with a small amount of a disubstituted amine having the formula—



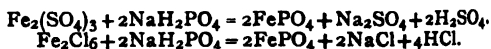
A better yield of the latter substance is obtained by prolonged boiling in alcoholic solution of one molecular proportion of the Compound I. with a second molecule of dimethyldihydroresorcin. Chloroketodimethyltetrahydrobenzene condenses with two molecules of a primary diamine to give a hydrochloride having the following formula:—



The corresponding base is strongly alkaline to litmus, whereas the Compound I. has a neutral reaction; it cannot be made to condense further with dimethyldihydroresorcin, as, owing to its strongly basic nature, it is precipitated by this substance from an alcoholic solution in the form of an insoluble resorcin salt having the formula $\text{C}_{20}\text{H}_{24}\text{N}_4\cdot\text{C}_8\text{H}_{12}\text{O}_2$.

42. "A Modification of the Volumetric Estimation of Free Acid in the presence of Iron Salts." By C. CHESTER AHLUM.

Because of the inapplicability of indicators in the titration of free acid in the presence of iron salts, the author devised a modification in which the iron is removed from solution, making a titration possible. The iron is precipitated by means of sodium dihydrogen phosphate. The iron phosphates are filtered off, and the filtrate titrated with standard sodium hydroxide. In the reaction of the ferric salt with the sodium dihydrogen phosphate, a definite quantity of acid is liberated, necessitating a correction in the amount of acid found in the titration.



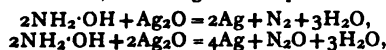
The amount of acid is directly proportional to the amount of ferric iron present, the latter being estimated previous to the titration. The difference between the amount of acid found in the titration and the amount of acid equivalent to the ferric iron present gives the true amount of free acid. A table giving the results of a number of estimations of free acid in mixtures of known quantities of ferric sulphate and sulphuric acid shows a considerable degree of accuracy.

Calcium, magnesium, and ferrous salts do not interfere with the estimation, and the method is applied to the analysis of natural waters containing iron salts and free acid.

43. "The Theory of Alkaline Development, with Notes on the Affinities of certain Reducing Agents." By SAMUEL EDWARD SHEPPARD.

The author first dealt with the reactions of hydroxylamine and hydrogen peroxide respectively with silver salts, with notes on the reactions of organic reducers. It was shown that at moderate concentrations one molecule of

hydroxylamine reduces one molecule of silver salt, but at great dilutions two, according to the equations:—



whilst one molecule of hydrogen peroxide reduced one molecule of silver salt, probably according to the equation—



hydrogen being one of the reaction products. The bearing on the molecular condition in solution of these substances was discussed. With organic reducers, such as polyphenols and aminophenols, a peculiar case of "coupled" reaction was examined, wherein the mutual presence of organic compound and sulphite retarded the total oxidation. The author has also investigated the dynamics of development with these reducers.

44. "Resolution of 2 : 3-Dihydro-3-methylindene-2-carboxylic Acid into its Optically Active Isomerides." By ALLEN NEVILLE.

When ethyl benzylacetoacetate is treated with concentrated sulphuric acid, 3-methylindene-2-carboxylic acid is formed, which on reduction with sodium amalgam gives 2 : 3-dihydro-3-methylindene-2-carboxylic acid. This acid forms with *l*-menthylamine a well-defined crystalline salt, which on crystallisation from ethyl acetate gives, after a few crystallisations, the pure salts of the *d*-acid and *l*-base. This substance crystallises in long white needles, melts at 170°, is easily soluble in all the ordinary organic media except ethyl acetate and ether, and has $[\alpha]_D + 27.35^\circ$ and $[M]_D + 89.63^\circ$. On decomposing this salt, *d*-2 : 3-dihydro-3-methylindene-2-carboxylic acid was obtained, which crystallised in long flat needles melting at 86°. It was insoluble in water, but soluble in all the ordinary organic media; it gave in alcohol $[\alpha]_D + 67.28^\circ$ and $[M]_D + 118.41^\circ$, and in benzene $[\alpha]_D + 76.86^\circ$ and $[M]_D + 135.27^\circ$. The sodium, potassium, barium, silver, and lead salts and the methyl ester were prepared.

From the more readily soluble portions of the menthylamine salt, the levorotatory acid was isolated. This was exactly similar in specific rotation and other properties to the corresponding dextrorotatory isomeride.

NOTICES OF BOOKS.

A Manual of Pharmacology. By WALTER E. DIXON, M.A., Cantab., M.D., B.S., B.Sc. Lond., D.P.H. Camb., &c. London: Edward Arnold. 1906.

THE plan adopted by the author of this work is excellent, and its use will do much to lighten the labours of the medical student by lessening the tedious memory work, which has hitherto been a stumbling block to many. The book gives a full general account of pharmacology, assuming that the reader possesses a fair working knowledge of physiology and chemistry. The drugs are classified on a basis partly chemical and partly physiological, and the source and chemistry of each drug is briefly discussed, while its action and effects are fully described and illustrated; notes are also added on the pharmacopoeial preparations, doses, &c. Only a very small amount of therapeutics is introduced, and that merely to illustrate various points in pharmacology. The manual is thoroughly satisfactory in every respect, and deserves to be widely used by medical students and practitioners.

Einführung in die Thermodynamik auf energetischer Grundlage. ("Introduction to Thermodynamics based upon Energetics"). By Dr. JULIUS MEYER. Halle-a-S.: William Knapp. 1905.

In this work the theory and results of thermodynamics, or rather such results as are of practical importance, for the book makes no attempt to cover the whole ground of the

science, are clearly and simply explained; the discussion of energy, which is represented as a reality and not an abstract conception, being particularly satisfactory. A short mathematical introduction gives a sketch of the meaning and use of differential coefficients and of integration, sufficient at any rate for a clear understanding of the earlier parts of the book, although the student might find it desirable to supplement the knowledge he acquired from the book with the help of some treatise on the calculi, in order to profit fully by such mathematical analysis as introduced in the later sections. We can recommend this book for the use of chemists to pave the way for the more difficult text-books in thermodynamics; especially in the earlier parts it describes methods and results in clear language, and very full explanations are given wherever difficulties are likely to occur to the beginner.

Etude Générale des Sels. ("General Study of Salts"). By ALFRED DITTE. Vol. I. and II. Paris: H. Dunod and E. Pinat. 1906.

THESE two volumes contain an exhaustive account of our knowledge of the subject of salts, special stress being laid upon defining and comparing the properties of groups rather than of individual members of each group. Vol. I. deals with the Binary Salts, and gives full details as to their preparation, chemical and physical properties, and the action of heat and electricity upon them, while copious tables are given of heats and formation, the author believing in the importance of emphasizing thermo-chemical facts. The second volume deals with the oxygenated Ternary Salts, and opens with a discussion of their general properties, and the action of various chemical reagents upon them; in the chapter on the action of water upon salts the theory of ionisation in dilute solutions is briefly considered, while the chapter on the reciprocal actions of acids, bases, and salts, and that on the action of acids upon salts, contain much interesting theoretical matter, clearly written and well arranged. In the last part of the volume the general principles are applied to the various groups of ternary salts. The book is one which a student will do well to consult for those important generalisations which are of such great value in showing him how to find his way along the paths in the forest of inorganic chemistry, as Prof. Ostwald puts it, while all the theoretical parts of the book are characterised by lucidity and admirable judgment, and the reader will find that he has nothing to unlearn when he passes on to the study of higher treatises.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

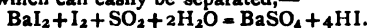
Comptes Rendus Hebdomadaires des Séances de l'Académie des Sciences. Vol. cxlii., No. 5, January 29, 1906.

New Researches on Insoluble Alkaline Compounds contained in Living Vegetables.—M. Berthelot.—The existence of insoluble potassium compounds and those of other alkaline metals in dead leaves, &c., and their derivatives, is of great interest, on account of the changes and migrations of this class of compound at different periods of vegetation and in the different organs of the plant. The author, in the present paper, describes a series of experiments executed on an oak tree.

Sulphates of the Rare Earths.—Camille Matignon.—The author prepares anhydrous sulphates of lanthanum, praseodymium, neodymium, and samarium. He starts with precipitated oxalates, which are carefully calcined at 800°. The oxide thus prepared is mixed with a slight excess of sulphuric acid, and the whole brought to a dull

red heat. Having prepared the pure anhydrous sulphates, the author finds the heat of solution. He also finds the heats of solution of the oxide in sulphuric acid. He concludes from the results obtained that the oxides of the rare earths from the point of view of their basic function are intermediate between the alkaline earth oxides and those of magnesium.

Rapid Preparation of Solutions of Hydriodic Acid.—F. Bodroux.—In ordinary, to prepare solutions of hydriodic acid with great rapidity a certain weight of iodine is divided into two equal parts. The first is treated with barium dioxide in presence of water, and is transformed into barium iodide, $\text{BaO}_2 + \text{I}_2 = \text{BaI}_2 + \text{O}_2$. The second portion of iodine is dissolved in the liquid thus obtained. A current of sulphurous anhydride is then allowed to pass through the liquid until decoloration just takes place. Hydriodic acid and barium sulphate are formed, which can easily be separated,—



An Alloy of Thorium and Aluminium.—O. Hönlischmid.—The reduction of thorium oxide by means of silicon in the electric furnace, as well as the direct combination of the silicide and thorium in presence of aluminium in a vacuum at 1000° , and finally the reduction of a mixture of potassium fluosilicate and the double fluoride of thorium and potassium by means of aluminium, are a means of preparing a crystalline thorium silicide in quadratic plates. These crystals very much resemble pure graphite, and have a composition corresponding to the formula ThSi_2 . Under analogous conditions the reduction of thorium oxide by means of aluminium in the electric furnace, the direct combination of aluminium and thorium in a vacuum, as well as the reduction of the double fluoride of potassium and thorium by means of aluminium, allow of the preparation of an alloy of thorium and aluminium crystallising in long hexagonal prismatic needles, with colour and metallic lustre resembling aluminium, and having a composition corresponding to the formula ThAl_3 .

Researches on the Halogen Compounds of Barium and Strontium Borates.—L. Ouvrard.—The borates of barium and strontium seem to enter into combination with chlorine and bromine less easily than the corresponding calcium salt. In both the former cases the author has only succeeded in obtaining a single halogen compound. In the case of the alkaline earth iodides the very slight stability seems to prohibit them entering into combination with the borates of the same metals, at least under the conditions of the author's experiments.

α - and β -Campholytic Alcohols.—G. Blanc.—The present research completes the list of compounds which the author has been obtaining for several years past by the reduction of β -campholytic ether under different conditions. He now prepares:—(1) β -campholytic alcohol by the reduction of the ether by means of sodium in aqueous medium; (2) β -dihydrocampholytic alcohol by the reduction of the ether by means of sodium and alcohol with a yield of 60 per cent; (3) α -campholytic alcohol either by the reduction of the ether or by the reduction of the amide by sodium and alcohol.

MEETINGS FOR THE WEEK.

- MONDAY, 5th.**—Royal Institution, 5. General Monthly Meeting. Society of Chemical Industry, 8. "Ignition of Nitro-compound Explosives in Small Arm Cartridges," by W. D. Borland.
- TUESDAY, 6th.**—Royal Institution, 5. "Food and Nutrition," by Prof. William Stirling, M.D., &c.
- Society of Arts, 4.30. "Imperial Questions in the West Indies," by Sir Neville Lubbock, K.C.M.G.
- WEDNESDAY, 7th.**—Society of Arts, 8. "The Artistic in Painting and Photography," by J. C. Dollman.
- Society of Public Analysts, 8. "Simple and Rapid Method for the Approximate Estimation of Boric Acid," by C. H. Cribb and F. W. F. Arnaud. "Analysis of a Sample of Air extinguishing a Flame," by B. Blount. "Detection of Coconut Oil in Butter," by A. W. Thorp. "Composition of Milk," by H. Droop Richmond.

- THURSDAY, 8th.**—Royal Institution, 5. "The Physiology of Plants," by Francis Darwin, M.A., &c.
- FRIDAY, 9th.**—Royal Institution, 9. "Some Dietetic Problems," by Robert Hutchinson, M.D., &c.
- Physical, 8. "Velocities of the Ions of Alkali Salt Vapours at High Temperatures," by Prof. H. A. Wilson. "Some Experiments on Earth-currents at Kew Observatory," by Dr. Harker.
- SATURDAY, 10th.**—Royal Institution, 3. "The Corpuscular Theory of Matter," by Prof. J. J. Thomson, F.R.S., &c.

NOTES AND QUERIES.

* Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Nitro-starch—Can any reader supply information relating to the recent bibliography of Nitro-starch?—J. W. G.

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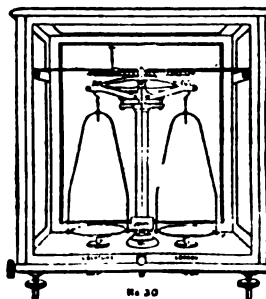
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ON THE ESTIMATION OF INDUSTRIAL TARTARIC ACID.

By Dr. P. CARLES.

A MERCHANT has made the following experiment: Wishing to buy a large quantity of tartaric matters "mixtes" (by this word we mean the compound of tartrate of lime and bitartrate of potash) he has taken a sample on the lot with the precaution required in such a case, and that sample he has divided into six similar small ones, numbered from one to six. Two have been sent to Mr. X., an Italian chemist, specialist for tartaric matters, two others to Mr. Y., a French chemist, specialist in the same line, and the last two to Mr. Z., another French chemist, also a specialist.

Each of the three, ignorant of the similitude of the two parcels, gave the same figures for the two samples he analysed, which constitutes a good mark for all. But between X. and Y. there was a difference of nearly 2°, between Y. and Z. almost the same, so that between X. and Y.'s figures the difference was nearly 4°.

Persons knowing the market value of those matters will certainly be moved in reading the above results, and still more when they come to think of the large quantity of goods involved.

What can be the causes of all three chemists being at variance? To ascertain them we will successively examine the operations most aleatory of the Goldenberg and Geromon 1898 method. That method had been prescribed to the three experts.

Chlorhydric Acid.—Differences in density in the acid used are of small importance in this case, for the tartrates in a very fine powder are easily soluble in the acid, especially when diluted.

Gauging.—The author of this method has been careful to recommend to ascertain that the 50 c.c. measure is really half of the 100 c.c. one, volume given to the chlorhydric solution. It is most probable that nobody forgets that verification.

Transformation of the Tartrate of Lime into Neutral Tartrate of Potash.—This is, in our opinion, one of the points on which it is easiest to make a mistake. When, as is usually done, one pours the carbonate of potash into the chlorhydric liquor, a precipitation of the lime salts very often takes place, and that precipitation is the more rapid and abundant as the tartrate of lime occupies more room in the mixture. Now, although at our instigation Mr. Goldenberg has recommended in 1889 to be sure that the liquid remains alkaline until the end, nothing indicates with precision, after ten minutes ebullition, that the conversion of the tartrate of lime into potassic tartrate is complete. That is why, when the raw material is rich in tartrate of lime, if the washed precipitate is placed in contact with acetic acid, one sees that one part of it does not dissolve, and that it is tartrate of lime.

It is to obviate that state of things that we have proposed to pour, not the carbonate of potash into the chlorhydric solution, but to do the reverse, slowly and *à froid*, boiling afterwards the mixture for twenty-five minutes instead of ten. That modification offers the following advantages:—

1. With the first drops of acid liquor the carbonate of potash is transformed into bicarbonate.
2. This bicarbonate does not precipitate the salts of lime, for when the last drops of acid are poured all is invariably in dissolution.

3. When warming this liquid, without ceasing to agitate with the hand, the bicarbonates dissociate, the carbonic gas disengages itself with the speed that one wishes; and of the salts of lime separated by ebullition not one particle escapes the action of the carbonate alkaline.
4. Those particles are so small that the ebullition takes place quietly until the end and without any of those disquieting jumps which occur when one proceeds as said further above.
5. The conversion of tartrate of lime is complete, as everyone can verify in the way already described.

Transformation of the Bitartrate.—The separation of the neutral tartrate of potash, the washings, the evaporation to a nicety of the liquor and the transformation of the tartrate neuter into bitartrate by acetic acid carry no serious cause of error.

Washing of the Bitartrate.—Some make, by dilution in the capsule itself, the ten or twelve washings with alcohol necessary to extract the adhering acetic acid, and only at the end do they pass the precipitate through the conical paper. Others prefer to do all the washings on the filter itself by "lixiviation." We have always considered the first method as the better, because when the matter is gummy or "pectineuse," the alcohol passes without penetrating to the centre of the cone, which retains then its acetic acid.

Dissolution of the Bitartrate.—The author advises to pass it into a glass or conical vase and to dissolve it with the warm water used in rinsing the capsule. But it seems to us more prudent to replace it in the capsule, where the alkaline reaction of the glass is not to be feared, unless those glasses have been boiled a long time with chlorhydric water, a prudent method when they are to be kept specially for tartaric experiments.

Titrated Alkaline Solution.—Everybody agrees it should only be formed of soda free from carbonate, but one should not forget that, to our instigation again, Mr. Goldenberg has recommended to standardise it, to use bitartrate pure and not oxalic or sulphuric acid themselves standardised with care, such as was generally done scarcely fifteen years ago.

Standard Pure Bitartrate of Potash.—We affirm that it is not as easy as is commonly thought to obtain a salt worthy of that name, and yet its purity is here of capital importance. Some pretend to arrive at it by putting some cream of tartar (ground to a fine powder) to digest in some chlorhydric water, and by washing afterwards in distilled water. But we humbly confess that after using 500 of chlorhydric water at one-tenth to purify 400 of cream of tartar rich, and washing afterwards with 2 litres of water, we have only obtained, in spite of a loss of 20 per cent, a bitartrate at 99.35 per cent. We therefore believe absolutely necessary to prepare that standard bitartrate by synthesis as follows:—

In a litre of distilled water, dissolve when warm 100 of tartaric acid, filter, and divide into two equal parts. In one of them, boiling, add a solution made when cold of carbonate of potash pure in distilled water, and filtered until cessation of effervescence. Then add the tartaric acid kept in reserve slowly and without ceasing to stir with a glass spatula. Leave to cool, decant, and wash the deposit with warm distilled water. The product desiccated is pure, but is hygroscopic. Re-dissolve it in boiling distilled water, and decant the solution into other porcelain capsules. Next day separate the *eaux mères*, wash with distilled water, drain, and dry until constant weight in a water-bath.

You have then a perfectly reliable standard worthy of confidence to titrate the "sodique" liquor.

Test-tubes.—We have long ago said, with others, how unreliable are the instruments sold by the trade. The test-tubes are among that number. It is indispensable to always reserve the same. 1° to titrate the sodique liquor, 2° to titrate the bitartrate separated from the Goldenberg.

When one comes to think of the differences produced on the value of a waggon of goods by a few tenth of degrees of that instrument, one finds that the precaution is worth taking.

Indications.—Goldenberg advises the use of turnsol paper; others think that phenolphthalein indicates with more accuracy the limit of saturation. We are among these, but with one absolute condition, as with in fact all witnesses, that one operates with the bitartrate unknown in the same manner as with the standard pure bitartrate; using as much as possible the same weights of matter, an equal volume of water at the same temperature, and an equal number of drops of the indicator.

Such are the observations that we have noted during a long practice of this method of dosing the tartaric acid in the tartaric matters on the market. That method, saving a few exceptions, gives, without a doubt, perfect results. But the details given above tell clearly how necessary it is to have practiced it long, for as you have just read, the method admits of causes of errors both ways.

THE DISSOLUTION OF PLATINUM BY SULPHURIC ACID.

By MARCEL DELÉPINE.

If we consult text-books, papers, or dictionaries of chemistry as to the action of sulphuric acid with regard to platinum, it becomes very difficult to form an opinion. Of twenty-one French and German works, of which the oldest dated back only to 1880, five stated that platinum is attacked slightly or slowly by sulphuric acid when heated, seven that it is not attacked, and nine that it is only attacked by a nitrous sulphuric acid. These divergent opinions, without doubt, have their origin in the papers by Scheurer-Kestner on this subject.

After having shown that the commercial platinum apparatus used for concentrating sulphuric acid are corroded in every part, the more so as the concentration is pushed further (Scheurer-Kestner, *Bull. Soc. Chim.*, 1875, [2], xxiv., p. 501; 1878, xxx., p. 28; *Comptes Rendus*, lxxxvii., p. 1082), Scheurer-Kestner returned to the subject, and announced that the solution of the platinum depended upon the presence of nitrous products; that a pure acid free from nitrogen does not attack platinum (*Comptes Rendus*, 1880, xci., p. 59).

Recently M. Conroy (*Journ. Soc. Chem. Ind.*, 1903, xxii., p. 465) carried out further experiments by heating strips of platinum-foil in sulphuric acid to about 250° to 280°, and he entirely refuted Scheurer-Kestner's results. At these temperatures, which are those of the acid in the retorts during distillation, the platinum is always attacked; different substances influence the progress of the attack; nitrous acid, and especially arsenious acid, have a retarding action; most other substances likely to be met with in commercial acids, such as SO_4Pb , SO_4Fe , NaCl , $\text{SO}_4(\text{NH}_4)_2$, &c., have either slightly accelerating actions, or none at all.

My experiments on the decomposition of sulphate of ammonium have led me to complete this research from the temperature of boiling pure sulphuric acid (338°), and that of the acid more or less charged with sulphate of potassium; I have given the results, not by the volume of acid employed, but in a uniform manner of one hour's attack on a surface of 1 square decimetre (50 sq. c.m. on each side of a sheet of platinum-foil); this valuation alone is logical, as it is evident that the attack must be proportional to the surface. I must here remark that from one experiment to another the results may vary considerably according to the rhythm of the boiling, which is impossible to regulate. The strips of platinum used were from 10 to 20 μ in thickness; they consisted of ordinary commercial platinum; during the course of the experiments they lost more than half their weight, and became very friable.

The attack of a strip of platinum in a porcelain or platinum crucible only takes place to a very slight extent; but if we put the same strips and acid into a flask and boil for half-an-hour, we observe that the acid becomes yellow, and gradually the colour becomes deeper until it takes that of red ochre. Under these conditions a square decimetre loses about 0.01 grm. per hour (0.008 to 0.012 grm.); if the attack were uniform the loss of thickness on each side would be about 0.05 μ ; in practice the metal is attacked unequally, and takes a dull appearance. The difference of the action, according to whether we operate in a wide open vessel like a crucible or a narrow one like a flask, is due entirely to the temperature, which only reaches 250° to 270° in the first case, because the acid evaporates so quickly, while it goes up to 338° in the second case, where the evaporation is very slight.

The influence of temperature is, in fact, considerable. At 350° to 355° (the boiling-point of 50 grms. SO_4H_2 + 10 grms. SO_4K_2) the loss of weight reaches as much as 0.04 to 0.05 grm., and at 365° to 370° (the boiling-point of 50 grms. SO_4H_2 + 20 grms. SO_4K_2) it goes as high as 0.12 to 0.13 grm. In this latter case, with a square decimetre of surface to be attacked, a few minutes is sufficient to cause the acid to take a yellow colour.

These results were obtained with pure commercial acid having no action on ferrous sulphate, which, when used as recommended by M. Berthelot in his lectures, will indicate the presence of 1/50,000th to 1/100,000th part of nitrous compounds, but such an acid reacts on sulphate of brucine, and will show the presence of a few millionths of these compounds.*

This amount appears to be insufficient to explain the solution of the platinum by catalytic action, and to remove all doubt in this respect I undertook a special research.

First of all, I freed the acid from its nitrous products by diluting it to a density of 1.4 and re-concentrating it several times; according to Fresenius this operation is sufficient, and, in fact, acid thus diluted and boiled, when treated with sulphate of brucine, gave no rose colour at all, or at most a tint so faint that the addition of a ten-millionth part of nitric acid made it incomparably more distinct. Now this acid, practically free from nitrous products, attacks the platinum strips in exactly the same manner as the original acid (0.008 grm.); when enriched by 1/50,000th, 1/25,000th, 1/10,000th, and 1/1000th part of nitric acid, it caused losses of platinum of 0.0075 grm., 0.0118 grm., 0.0083 grm., and 0.008 grm. respectively. If the nitrous products play a fermentary part, as thought by Scheurer-Kestner, we should expect to see the losses increase in the same proportion. In the same manner, by adding an excess of nitric acid to mixtures containing sulphate of potassium the speed of attack was not changed (0.133 grm. instead of 0.133 and 0.124 grm.).

On the other hand, sulphate of ammonium exercises the retarding action observed by Scheurer-Kestner in a most perfect manner. M. Conroy found this salt to be without any special action, but he operated at about 250°, and his acid was too slightly enriched with platinum on account of having been heated for an insufficient time for the retarding action to be observed. This effect at 338° is such that after obtaining a slight solution, the metal no longer changes weight so long as there are still a few m.grms. of ammonia per 30 c.c. of acid, then the solution takes its normal course. This is all easily explained by the following experiment:—If to a sulphuric solution of platinum we add sulphate of ammonium and then boil, the metal (and not platinum-black) is precipitated almost integrally in the form of sponge; there are certain restrictions and special

* A sulphuric solution of sulphate of brucine at 0.20 grm. per cent is a very sensitive reagent for nitrous and nitric acids. A drop of water containing one millionth part of nitric acid, placed on a drop of this solution, takes a bright red colour round its periphery. Two c.c. mixed with 1 c.c. of Paris water gave a bright red colour which changed to bright yellow in twenty-four hours.

† Sometimes it even increases a little on account of a slight deposit of platinum from the solution.

phenomena due to the formation of solutions with different properties, which I hope to examine later on.

This precipitated metal which is deposited on the surface of the strips of platinum when they are present, then exercises its decomposing action on the sulphate of ammonium without the compact metal being sensibly affected.

If we operate in the presence of sulphate of potassium, we also observe the retarding effect, but not a total stoppage as with the acid alone. Thus with 20 grms. SO_4K_2 + 50 grms. SO_4H_2 , the losses of platinum per hour, in the presence of a little sulphate of ammonium, were successively 0.0230 gm., 0.0210 gm., 0.0175 gm., 0.0406 gm., and 0.0901 gm. instead of 0.13 gm. These experiments also do away with the necessity for the presence of nitrous compounds, sulphate of ammonium appearing to be the agent most apt to destroy them.*

If, instead of compact platinum in strips, we take spongy platinum, we can dissolve it entirely. One gm. of sponge from the calcination of the chloroplatinates of aniline, quinine, and ammonia at a red heat, lost respectively in boiling sulphuric acid in the course of the first hour 0.06 gm., 0.033 gm., and 0.033 gm. If we compare these results with those obtained with strips of platinum we must remember that the surface of attack in 1 gm. of the two latter calcined sponges hardly exceeds 3 to 4 square decimetres; from this we can calculate the effect of the two extremes—as a surface folded as much as possible or else as a collection of little balls. In the first we should have a strip of about 2 μ thickness, in the second case the gm. would be composed of 170 million spheres each of 8 μ diameter. These figures are evidently only rough approximations deduced from the action of the sulphuric acid, but they lead us to the idea that reciprocally a strip of platinum sufficiently thin might perhaps fill the catalytic rôle of sponges of which the calculated thickness is relatively considerable. As a matter of fact, strips of commercial platinum of about 0.16 μ in thickness, slightly crumpled, will inflame hydrogen, will at the ordinary temperature change a solution of formic aldehyde into carbonic anhydride, and will make a flameless lamp with organic vapours, ammonia, &c., almost as well and conveniently as spongy platinum itself. As for the reaction of the attack it is practically $4\text{SO}_4\text{H}_2 + \text{Pt} = (\text{SO}_4)_2\text{Pt} + 2\text{SO}_2 + 4\text{H}_2\text{O}$; a platinum salt is formed, easily recognised by the addition of chloride of potassium to sulphuric solutions diluted with one volume of water; the liquid is decolourised, and deposits only octahedra. Still, the amount of SO_2 formed, compared with the loss of platinum, leads us to suppose that a little more platinum (1/20th) is dissolved than agrees with the above formula.—*Bull. Soc. Chim.*, Series 3, vol. xxv., No. 1.

WET AND CRUCIBLE-FIRE METHODS FOR THE ASSAY OF GOLD TELLURIDE ORES, WITH NOTES ON THE ERRORS OCCURRING IN THE OPERATIONS OF FIRE ASSAY AND PARTING.†

By W. F. HILLEBRAND and E. T. ALLEN.
(Continued from p. 101).

PART I. ASSAY OF CRIPPLE CREEK TELLURIDE ORES (continued).

Fire Assay.

Conduct of the Assay.

The assays were all made in Battersea F crucibles, heated in a gas-fed melting furnace, as the muffle available was

far too small to admit crucibles of such size. A very satisfactory charge was found to be that given below, and it was adhered to throughout, except in a few cases where the litharge was reduced to four assay tons without any apparent difference in results.

Ore	1 assay ton (29.166 grms.)
Sodium bicarbonate	1 assay ton
Litharge	6 assay tons
Borax (fused)	10 grms.
Salt cover	

Neither iron nor nitre was used. In most cases the requisite amount of silver was added to give two and one-half to three times the weight of the gold. During the earlier stages of firing the temperature was purposely kept low, and it was not made high until escape of gas had nearly ceased. The buttons obtained were rather large for cupellation—about 25 grms. in weight—but it was felt that, by reducing a large amount of lead, the collection of all the gold would be more certain. The buttons were in all cases soft and malleable. Not every slag was assayed, since it was found that the gold losses here were very slight, in direct disagreement with Fulton's results on Cripple Creek tellurides (*Journ. Am. Chem. Soc.*, 1898, xx., 596; see also *prox.*, "Discussion of Results").

The larger number of tests were made by one of us (A.), but in order to have the check of another worker a number were made by his colleague (H.). The latter's results seem to be in general a little below those of A.—a fact probably explained by the employment of a somewhat higher temperature in cupellation. The powerful influence of slight changes in temperature on the cupellation results will be emphasised in the second part of this paper.

Charges Used in Assaying Slags and Cupels.

The charge used for the slag assay was:—Litharge, 1 A.T.; argol, 2 grms.; salt. For cupels it was:—Litharge, 2 A.T.; soda, 1 A.T.; borax glass, 1.5 A.T.; argol, 2 grms.; salt.

Assays for Gold and Silver together.

The first assays were made in order to ascertain, not only the gold, but also the silver content of the ores, though no importance was attached to the determination of the latter. Incidentally these tests showed the losses of gold in slags and cupels.

Discussion of Results.—The cupellation loss, even without that due to volatilisation, is here seen to be markedly greater than in the slag. This observation is in agreement with the general tenor of statements found in the literature, but directly contradicts Fulton's statement, already referred to, as to telluride ores from Cripple Creek (*Journ. Am. Chem. Soc.*, 1898, vol. xx., p. 596). May not his high losses in the slag have resulted from his employment of iron nails, with resultant formation of auriferous sulphide in the slag?

In order to have a check upon the actual loss, including that due to volatilisation, proof metal of the composition of that obtained from the ore was cupelled with 25 grms. of lead. The results are given in Table IV.

When cupellation takes place at a low temperature, with formation of considerable feather litharge, as in the above cases, the loss by volatilisation is seen to be practically negligible, or if not absolutely inconsiderable to be compensated perhaps by retention of lead, but that the case is different when the temperature is higher is plainly shown elsewhere in this paper (Tables VIII. and IX.).

Assays for Gold alone.

In all these cases the requisite amount of silver was added at the start to give a bead suitable for parting. (See Table V.).

In order to check the actual loss, including that by volatilisation, cupellations were made with alloys corre-

* It is not absolutely certain that the last few millionths are destroyed when we distil the acid with a few thousandths of sulphate of ammonium; at least this is so according to an experiment I made; both the distillate and the residue coloured sulphate of brucine. It remains to be seen, however, whether the very feeble colouration observed is really due to nitric acid.

† United States Geological Survey. *Bulletin* No. 253. (Series E, Chemistry and Physics, 44).

sponding to the above and about 25 grms. in weight. (See Table VI.).

When the above percentage recoveries are compared with corresponding ones for the alloy far richer in gold (Table IV.), it is seen, as might be expected, not only that

TABLE III.—*Crucible Assays of Cripple Creek Telluride Ores for Gold and Silver together.*

(Values are the weights obtained in m.grms., and denote ounces per ton of 2000 pounds).

Main assay, Au + Ag.	Slags.		Cupels.	
	Au + Ag.	Au.	Au + Ag.	Au.
a... ..	16.54	—	—	—
	16.37	0.07	—	—
	16.97	0.04	—	—
	16.50	—	—	—
	16.58	—	—	—
	16.54	—	—	—
Average ..	16.58	0.055	—	0.085
From slag ..	0.055	—	—	—
From cupel ..	0.085	—	—	—
Total ..	16.72	—	—	—
Less average gold from Table VII.	14.53	—	—	—
Silver ..	2.19	—	—	—
b	20.54	—	—	—
	20.63	0.08	—	—
	20.58	0.08	0.03	Lost
	20.88	—	0.10	0.04
	20.21	—	—	—
	—	—	—	—
Average ..	20.57	0.08	0.015	0.10
From slag ..	0.08	—	—	0.04
From cupel ..	0.10	—	—	—
Total ..	20.75	—	—	—
Less average gold ..	17.75	—	—	—
Silver ..	3.00	—	—	—
a ₁	17.39	0.43	—	Lost
	17.40	0.05	—	0.17
	17.43	0.07	—	0.15
	17.25	0.03	—	0.17
	—	—	—	—
	—	—	—	—
Average ..	17.37	0.145	0.027*	0.16
From slag ..	0.145	—	—	0.7*
From cupel ..	0.16	—	—	—
Total ..	17.675	—	—	—
Less average gold ..	15.60	—	—	—
Silver ..	2.075	—	—	—
b ₁	22.33	0.06	—	0.22
	22.34	0.05	—	0.17
	22.42	0.06	—	0.16
	22.38	0.04	—	0.18
	—	—	—	—
	—	—	—	—
Average ..	22.37	0.05	0.027*	0.18
From slag ..	0.05	—	—	0.07*
From cupel ..	0.18	—	—	—
Total ..	22.50	—	—	—
Less average gold ..	19.63	—	—	—
Silver ..	2.87	—	—	—

* Averages obtained by combining all the beads (8) from a₁ and b₁.

TABLE IV.—*Cupellation Losses on Proof Alloy.*

	Taken.	Found.	Loss.		Recovered from cupels.	
			M.grms.	Per cent.	M.grms.	Per cent.
Ag ..	2.09	—	—	—	0.06	2.87
Au ..	15.59	—	—	—	0.077	0.49
Au + Ag	17.68	17.53	0.15	0.85	0.137	—
Ag ..	2.26	—	—	—	0.08	3.54
Au ..	15.74	—	—	—	0.077	0.49
Au + Ag	18.00	17.84	0.16	0.89	0.157	—
Ag ..	2.28	—	—	—	0.07	3.07
Au ..	19.75	—	—	—	0.08	0.40
Au + Ag	22.03	21.51	0.52	2.36	0.15	—
Ag ..	2.58	—	—	—	0.10	3.88
Au ..	19.64	—	—	—	0.07	0.36
Au + Ag	22.22	22.07	0.15	0.68	0.17	—

TABLE V.—*Crucible Assays of Cripple Creek Telluride Ores for Gold alone.*

(Values are the weights obtained in m.grms., and denote ounces per ton of 2000 pounds).

Main assay, Au.	Slags.		Cupels.	
	Au + Ag.	Au.	Au + Ag.	Au.
a	14.50	0.25	—	—
	14.42	0.36	—	—
	14.45	0.16	—	0.72
	14.47	0.18	—	0.82
	—	—	—	—
	—	—	—	—
Average ..	14.46	0.24	0.02	0.77
From slag and cupel	0.05	—	—	0.03
Total ..	14.51 (Allen).	—	—	—
a	14.63	—	—	—
	14.63	—	—	—
	14.53	—	—	—
	14.21	—	—	—
	—	—	—	—
	—	—	—	—
From slag and cupel	14.50	0.05	—	—
Total ..	14.55 (Hillebrand).	—	—	—
a ₁	15.50	0.08	—	0.92
	15.52	0.09	—	1.00
	15.55	—	—	1.23
	15.61	—	—	1.07
	15.57	—	—	—
	15.53	—	—	—
Average ..	15.55	0.09	0.04	1.05
From slag and cupel	0.07	—	—	0.03
Total ..	15.62 (Allen).	—	—	—
a ₁	15.50	*	0.02	*
	15.48	—	—	0.10
	—	—	—	0.03
	—	—	—	—
	—	—	—	—
	—	—	—	—
Average ..	15.49	—	0.02	—
From slag and cupel	0.08	—	—	0.06
Total ..	15.57 (Hillebrand).	—	—	—
b	17.76	0.32	—	0.87
	17.83	0.28	—	0.82
	—	—	—	—
	—	—	—	—
	—	—	—	—
	—	—	—	—
Average ..	17.79	0.30	0.02	0.85
From slag and cupel	0.05	—	—	0.03
Total ..	17.84 (Allen).	—	—	—

TABLE V. (continued).

Main assay, Au.		Slags.		Cupels.		
		Au + Ag.	Au.	Au + Ag.	Au.	
b		17'69	*	0'02	—	0'03
		17'73	*	0'02	—	0'03
		17'47	*	0'03	—	0'03
		17'14†	*	0'03	—	0'03
Average		17'63	—	0'025	—	0'03
From slag and cupel		0'055				
Total		17'68½	(Hillebrand).			
b ₁		19'39	0'14	—	1'48	0'05
		19'53	0'14	—	1'34	0'06
		19'51	—	—	1'28	0'03
		19'57	—	—	1'29	0'04
		19'55	—	—	—	—
		19'62	—	—	—	—
Average		19'54	0'14	0'04	1'35	0'045
From slag and cupel		0'085				
Total		19'625	(Allen).			
b ₁		19'79	*	—	—	—
		19'48	*	—	—	—
Average		19'63	—	0'02	—	0'05
From slag and cupel		0'07				
Total		19'70‡	(Hillebrand).			

† Proof metal (19.85 m. grms. gold and 49 m. grms. silver) was crucibled, impelled, and parted at the same time as these last two assays. The final gold was 19.84 + 0.07, or 19.91 in all, showing that in this case there must have been a marked retention of silver after parting. If a correction has to be applied to the regular assays, the average drops to 19.64, almost exactly that of Allen.

TABLE VI.—Cupellation Losses on Proof Alloy.

	Taken.	Found.	Loss.		Recovered from cupsels.	
			M.grms.	Per cent.	M.grms.	Per cent.
Ag ..	48·03	46·90	1·13	2·35	0·82	1·71
Au ..	15·79	15·70	0·09	0·57	0·02	0·12
Ag ..	55·37	54·77	0·60	1·08	—	—
Au ..	15·82	15·75	0·07	0·44	—	—
Ag ..	63·65	62·27	1·38	2·17	1·09	1·71
Au ..	19·72	19·63	0·09	0·46	0·02	0·10
Ag ..	63·81	62·46	1·35	2·11	1·04	1·63
Au ..	19·74	19·64	0·10	0·51	0·03	0·15

TABLE VII.—*Comparison of Results by the Two Methods.*

				Fire assay.	
				Hillebrand.	Allen.
<i>a</i>	..	..	14.76 ^a	14.55 (Average, 14.53†)	14.51
<i>b</i>	..	..	17.71	17.69 (Average, 17.75†)	17.84
<i>a</i> ₁	..	..	15.50	15.57 (Average, 15.61†)	15.62
<i>b</i> ₁	..	..	19.47	19.64 or 19.70 (Averages, 19.63† or 19.64†)	19.62 or 19.62†

† See footnote to Table V. for explanation of the alternative value.

(To be continued).

$$\text{C}_8\text{H}_{14} \begin{cases} \text{C} : \text{CH} \\ | \quad | \\ \text{CO} \quad \text{N} \end{cases} \begin{cases} \text{C}_6\text{H}_5 \\ \text{O}_2\text{SC}_6\text{H}_5 \end{cases}$$

* From the *American Chemical Journal*, xxxiv., No. 3.

Amines.	$\text{C}_8\text{H}_{14} \begin{array}{c} \text{C} : \text{CCOONH}_3\text{R} \\ \text{CO NH} \end{array}$	$\text{C}_8\text{H}_{14} \begin{array}{c} \text{C} : \text{COOH} \\ \text{CO NH} \end{array}$	$\text{C}_8\text{H}_{14} \begin{array}{c} \text{C} : \text{CCH} \\ \text{CO NH} \end{array}$
I. $\text{C}_{10}\text{H}_7 \cdot \text{NH}_2$ β	169°	*M. p. 172.5°	173°
II. $\text{C}_6\text{H}_4 \cdot \text{CH}_3 \cdot \text{NH}_2$ [1 : 4]	152°	168°	178°
III. $\text{C}_6\text{H}_5 \cdot \text{CH}_2 \cdot \text{NH}_2$	147.5°	140°	96.5°
IV. $\text{C}_{10}\text{H}_7 \cdot \text{NH}_2$ α	165°	*170°	—
V. $\text{CH}_3\text{C}_6\text{H}_4\text{NH}_2$ [1 : 3]	126°	154°	—
VI. $\text{NH}(\text{C}_2\text{H}_5)_2$	+139.5°	—	153°
VII. $\text{NH}(\text{CH}_3)_2$	+137.5°	—	63°
VIII. NH_2CH_3	172°	—	131°
IX. $\text{C}_6\text{H}_5\text{C}(\text{NH}_2) \cdot \text{NH}$	—	—	— (a)
X. $\text{NH}_2\text{C}_6\text{H}_4 \cdot \text{C}_6\text{H}_4\text{NH}_2$	—	—	— (b)
XI. $\text{CH}_3\text{C}_6\text{H}_3\text{NH}_2 \cdot \text{NO}_2$	—	—	192°
XII. $\text{NH}_2\text{CONHNH}_2$	200° and 209—210°	—	—
XIII. $\text{C}_6\text{H}_5\text{NH}_2$	*158°	*174°	*166°
XIV. NH_2OH	—	*+146.5°	—
XV. NH_3	—	*178°	—
XVI. $\text{C}_6\text{H}_4(\text{NH}_2)_2$ [1 : 2]	—	—	— (c)
XVII. $\text{NH}_2\text{NHC}_6\text{H}_5$	—	—	— (d)
XVIII. $\text{CH}_3\text{CONHNHC}_6\text{H}_5$	—	—	—
XIX. $\text{OC}(\text{NH}_2)_2$	—	—	—
XX. $\text{NH}_2\text{C}_6\text{H}_4\text{NO}_2$ [1 : 4]	—	—	—
XXI. $\text{CH}_3\text{COC}_6\text{H}_4\text{NH}_2$ [1 : 4]	—	—	—
XXII. $\text{C}_6\text{H}_5\text{NHCH}_3$	—	—	—
XXIII. $\text{C}_6\text{H}_5\text{NHC}_2\text{H}_5$	—	—	—
XXIV. $\text{C}_6\text{H}_5\text{NHCH}_2\text{C}_6\text{H}_5$	—	—	—
XXV. $\text{C}_2\text{H}_4(\text{NH}_2)_2$	—	—	—
XXVI. $\text{C}_6\text{H}_5\text{NHNHC}_2\text{H}_5$	—	—	—
XXVII. $(\text{NH}_2)_2\text{C} : \text{NH}$	—	—	—
XXVIII. $\text{CH} \begin{array}{c} \text{CH} \cdot \text{CH} \\ \text{CH} \cdot \text{CH} \end{array} \text{N}$	—	—	—
XXIX. $\text{C}_6\text{H}_5\text{N}(\text{CH}_3)_2$	—	—	—
XXX. $\text{CH}_3\text{C}_6\text{H}_4\text{NH}_2$ [1 : 2]	—	—	—
XXXI. $\text{NH}_2\text{NHC}_6\text{H}_4\text{Br}$ [1 : 4]	—	—	—
XXXII. NH_2NH_2	—	—	—

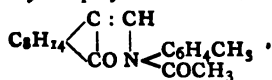
(a) $\text{C}_{19}\text{H}_{24}\text{N}_2\text{O}_4$. 184°.

(b) $\text{C}_8\text{H}_{14} \begin{array}{c} \text{C} : \text{CCOONH}_3 \\ \text{CO NH} \end{array} \text{C}_6\text{H}_4\text{C}_6\text{H}_4$. 190°.

(c) $+ \text{C}_{18}\text{H}_{20}\text{N}_2\text{O}_2$. 246°.

(d) $+ \text{C}_8\text{H}_{14} \begin{array}{c} \text{C} : \text{CCOOC}_2\text{H}_5 \\ \text{CO NHNHC}_6\text{H}_5 \end{array}$. 212°.

Acetyl chloride, when allowed to react with *p*-tolylcamphorformeneamine, replaces the iminic hydrogen and forms acetyl *p*-tolylcamphorformeneamine,—



which is a colourless crystalline compound melting at 161°. The reaction of these two chlorides with the above typical camphorformeneamines, resulting in the elimination of hydrochloric acid and the formation of the sulphone and acetyl derivatives, seems to be rather conclusive evidence in favour of the structure which has been suggested for the camphorformeneamines, and as we know that these bodies are formed from the corresponding carboxylic acids by the elimination of carbonic anhydride, the formulæ assigned to these acids also seem to be confirmed.

Chloracetyl chloride, when allowed to react with β -naphthylcamphorformeneamine, yields a colourless crystalline compound melting at 63°; unfortunately, on account of its instability, it could not be analysed.

The accompanying table shows the various condensation compounds of camphoroxalic acid and amines which have hitherto been prepared. They are arranged in type classes, and their melting-points are given. The compounds marked * have been previously described by J. Bishop Tingle and Alfred Tingle (*Journ. Am. Chem. Soc.*, 1901, xxiii., 364). Those marked + contain the elements of 1 molecule of water more than is shown in the type formula. Their constitution has been already discussed. In cases where no definite compound could be isolated, the space in the table is occupied by a dash.

EXPERIMENTAL.

Camphoroxalic acid was prepared according to the method worked out by J. Bishop Tingle (*Ibid.*, 1901, xxiii., 364) as follows:—

Camphor (31.5 grms.) and ethyl oxalate (22 grms.) were dissolved in 400 c.c. of anhydrous petroleum ether of 85–100° boiling-point and 3.5 grms. of sodium wire were added. The mixture was boiled on a water-bath with a reflux condenser until the violent action had ceased and the sodium had almost entirely dissolved; it was then allowed to remain over-night at the ordinary temperature, protected from moisture. The solution, after standing, was well shaken with about an equal volume of ice-water and the petroleum ether extract separated from the water and dried over calcium chloride. The dried liquid was distilled, at first from the water-bath; when the temperature of the boiling liquid rises to almost 90° the pressure is somewhat reduced and the distillation continued, care being taken not to allow the temperature of the liquid in the flask to rise above 130°; frequently, it is unnecessary to heat above 120–125°.

The aqueous solution was then well cooled and acidified with dilute sulphuric acid, then extracted three or four times with ether, until a portion of the fresh ether extract showed only a pale reddish violet colour with alcohol and ferric chloride. The ether was dried over calcium chloride, and, after filtering, was distilled off, and the residue added to that from the petroleum ether extract. This residue was extracted several times by boiling with a 10 per cent solution of potassium hydroxide until the filtrate, after acidification, no longer gave a marked colour with alcohol and ferric chloride. The alkaline solution from the filtered

extracts was then acidified with dilute sulphuric acid, and, after standing several hours, the crystalline precipitate was filtered off. This crystalline mass was purified by re-crystallisation from petroleum ether by means of a Soxhlet apparatus. The mother-liquors from the re-crystallisations and the residue in the Soxhlet apparatus were treated with sodium carbonate, the solution filtered, acidified, and the resulting precipitate re-crystallised.

CAMPHOROXALIC ACID DERIVATIVES.

Metallic Salts of Camphoroxalic Acid.

Copper Camphoroxalate.—The acid (1 molecule) and copper nitrate (0.5 molecule) were separately dissolved in 50 per cent alcohol, the solutions mixed and allowed to evaporate at the ordinary temperature. Although the pure and dry copper salt does not melt below 275°, it was found that, if the solution was heated on a water-bath to remove the excess of alcohol, the green salt formed with part of the solvent an oily layer which was extremely difficult to purify. When it is allowed to crystallise at the ordinary temperature, it is deposited as a fine, green, crystalline powder, which melts and decomposes at 275°. With ferric chloride in alcoholic solution this salt gives no colouration. If an alcoholic solution of the salt is heated for some time at 100°, it undergoes decomposition and deposits cuprous oxide.

Analysis.—2.1686 grms. salt gave 0.0474 grm. CuO.

Calculated for $C_8H_{14} \begin{matrix} \diagup C : COCOO \\ \diagdown CO \end{matrix} Cu$	22.28
Found	22.46

The proportion of the reacting substances was varied, in the hope that another salt of different composition might be formed, but in all cases the resulting compound was identical with the one described above.

2. Silver Camphoroxalate.—Silver nitrate and camphoroxalic acid were allowed to react in a solution of 50 per cent alcohol, and, on allowing the solvent to evaporate spontaneously, a colourless crystalline mass was obtained, which was purified by repeated re-crystallisation from alcohol. The resulting compound was found to melt and decompose at 137°.

Analysis.—0.2975 grm. salt gave 0.0965 grm. Ag.

Calculated for $C_8H_{14} \begin{matrix} \diagup C : COHCOOAg \\ \diagdown CO \end{matrix}$	32.63
Found	32.47

As in the preparation of the copper salt, different proportions of silver nitrate and camphoroxalic acid were allowed to react, but in this case also only one compound could be isolated.

3. Barium Camphoroxalate.—Barium nitrate (1 molecule) and camphoroxalic acid (2 molecules) in a solution of 50 per cent alcohol were allowed to evaporate. These deposited a colourless crystalline compound, which was re-crystallised from alcohol. With ferric chloride and alcohol a reddish violet colour, characteristic of the presence of the enol grouping, was produced.

Analysis.—0.1728 grm. salt gave 0.0682 grm. BaSO₄.

Calculated for $(C_8H_{14} \begin{matrix} \diagup C : COHCOO \\ \diagdown CO \end{matrix})_2Ba$	23.56
Found	23.21

The proportions of the reacting substances were varied, as in the preceding cases, but no other salt could be obtained.

4. Calcium Camphoroxalate.—Calcium nitrate (1 molecule) and camphoroxalic acid (2 molecules) were mixed in solution of 50 per cent alcohol, under the same conditions

as in the case of the barium salt; on evaporation and re-crystallisation from alcohol, colourless crystals were deposited.

Analysis.—0.2067 grm. salt gave 0.0537 grm. CaSO₄.

Calculated for $(C_8H_{14} \begin{matrix} \diagup C : COHCOO \\ \diagdown CO \end{matrix})_2Ca$	8.22
Found	8.4

In the case of the calcium salt also, only one compound could be obtained.

5. Ferric Camphoroxalate.—Ferric chloride (1 molecule) and camphoroxalic acid (2 molecules) were mixed in solution of 50 per cent alcohol. A deep reddish violet colouration was produced, but from no solvent could a crystalline compound be obtained, and the viscous substance deposited by the evaporation of the original liquid to dryness gave percentages of iron which showed that it had no definite composition; hence the following method was employed:—The alcoholic solution of the reaction-product formed by the camphoroxalic acid and ferric chloride was diluted with water, in order to precipitate any unchanged camphoroxalic acid, and, at the same time, to dissolve the dark red substance in solution in the alcohol. After filtering, ether was added to the liquid, but, on account of the presence of free hydrochloric acid which had been formed by the reaction of the camphoroxalic acid and ferric chloride, the ether dissolved and formed a homogeneous solution with the original liquid. When this mixed ethereal aqueous liquid was saturated with sodium chloride, the ether took up the red compound and separated from the aqueous layer; this latter was removed, and the ethereal solution dried over calcium chloride. The ether was then evaporated from the water-bath. A glassy almost black mass remained. It is soluble to a slight extent in boiling sodium carbonate solution. The compound should, probably, be regarded as being camphoroxalic acid, in which the hydrogen atom of the enol group :COH has been replaced by iron, while the carboxyl group remained intact on account of the presence of hydrochloric acid in the liquid during the reaction.

Analysis.—0.2095 grm. substance gave 0.0235 grm. Fe₂O₃.

Calculated for $(C_8H_{14} \begin{matrix} \diagup C : COCOOH \\ \diagdown CO \end{matrix})_3Fe$	7.72
Found	7.8

(To be continued).

PROCEEDINGS OF SOCIETIES.

PHYSICAL SOCIETY.

Ordinary Meeting, February 23rd, 1906.

Prof. J. PERRY, F.R.S., President, in the Chair.

A PAPER by Mr. J. WALKER entitled "A Note on Talbot's Lines," was read by the SECRETARY.

The diffraction-pattern of a line of monochromatic light seen in focus, due to a rectangular aperture with its sides parallel to the line, is characterised by dark bands arranged at equal intervals on either side of the geometrical image of the line. The effect of covering half of the aperture with a retarding plate is to displace the bands of an odd order towards the covered side by an amount proportional to the retardation introduced, those of an even order remaining fixed. Suppose that the light is white and that its monochromatic constituents have been made by spectral analysis to occupy different angular positions in the field. Owing to the dispersion, the bands of an even order are

obliterated; but in the case of those of an odd order the dispersing power of the plate itself produces a dispersion of the bands, and consequently these bands will be seen, provided the plate have a suitable thickness and be so placed that the dispersion of the bands produced by it acts in opposition to the primitive dispersion of the light. The *modus operandi* may be clearly seen by supposing the phenomena analysed by a spectroscope having its slit in the plane of the diffraction-pattern in a direction perpendicular to the bands. A simple calculation gives the "best thickness" of the plate.

The Secretary showed photographs of Talbot's lines forwarded to him by Mr. W. B. Croft.

A paper on "Secondary Röntgen Radiation" was read by Dr. C. G. BARKLA.

In previous papers the author has shown that the secondary X-rays from certain gases and light solids subject to Röntgen radiation may be fully explained by considering the corpuscles or electrons constituting the atoms to be accelerated in the direction of electric displacement in each primary Röntgen pulse as it passes through such substances, and that the interaction between the electrons affects only to a slight extent the character of the secondary radiation. The methods of investigating the more complex secondary radiation from metals was suggested by the results of the experiments on lighter substances. The character of secondary radiation (measured by absorbability) was found to depend on the atomic weight of the radiator. The radiation from compounds was in all cases such as would be given by a mixture of the radiations from the separate constituent elements. By heating an iron wire to white heat and studying the radiation proceeding from it when exposed to X-rays, it was seen that there was no direct connection between the intensity or character of the radiation and temperature, electric conductivity, or magnetic permeability. Experiments on absorption showed that the radiation from heavier atoms did not consist of an easily absorbed radiation superposed on a scattered radiation, such as proceeded from light atoms, but that the latter was not present to any appreciable extent.

No special absorption by a metal of the radiation proceeding from the same metal was observed. Thus there was no evidence of a definite periodic vibration. Changes in the character of the primary produced changes in the secondary. Variation in intensity of the primary beam was found not to affect the character of the radiation from calcium—the only substance experimented upon. When substances were exposed to polarised Röntgen radiation, the secondary radiations, which differed little in absorbability from the primary producing them, showed approximately equal variations in intensity in different directions. The very different radiations exhibited no such variation; while the radiation from a substance of atomic weight between those of the two classes showed the variation to an intermediate extent. In this polarisation effect compounds behaved as mixtures of the constituent elements. Experiments on the absorption of rays proceeding from thick plates of a large number of elements showed that beyond the region of atomic weights in which the character of the secondary radiation is almost independent of the nature of the radiation, the absorbability is a periodic function of the atomic weight of the radiator, and that as far as these experiments have gone different periods are represented by curves of similar form. The theory which has been found to explain all the phenomena of secondary radiation from light atoms may be extended to explain these results, if we conceive the independence of motion of the electrons to disappear with an increase in the number of electrons in the atom. Inter-electronic forces called into play make the pulses emitted by each electron thicker than if the electron were accelerated only during the period of passage of the primary pulse over it. Hence the radiation becomes less penetrating. Only agencies altering the construction of the atom would be expected to change the character of the radiation produced

by a given primary; hence, temperature, electrical resistance, magnetic permeability, may all vary without producing appreciable effect. The theory also explains the disappearance of the more purely scattered radiation, the absence of selective absorption, the connection and difference between the primary and secondary rays, and the absence of influence of variation in intensity of primary radiation on absorbability of the secondary. When applied to the atom as conceived by Prof. J. J. Thomson, the periodicity shown by the curve connecting absorption and atomic weight is explained.

A paper by Messrs. C. W. S. CRAWLEY and F. B. O. HAWES entitled "*Records of the difference of Potential between Railway Lines when a Train passes and at other times, and a suggested Method for the observation of Earth Currents and Magnetic Variations*," was read by Mr. CRAWLEY.

The experiments described in the paper were made on the London and South Western main line, between Walton and Weybridge stations. To each rail of the up line a wire was permanently attached, and the other ends of the wires were connected to the terminals of a reflecting galvanometer. The deflections of the galvanometer were recorded on a moving sheet of paper, and curves obtained showing the variation in the current through the galvanometer. The curves showed a concordance in the results from successive trains. The normal current through the galvanometer began to be disturbed about one minute before the passage of a train, and the disturbance lasted about two minutes. The curves, although very complex, showed some regularity. In addition to the disturbances caused by passing trains, very distinct smaller disturbances were noticeable at the times when a receding train was passing over the points at Walton and Esher stations. Effects were also shown corresponding with the passage of a train over the Hampton Court Junction points. The observations on different occasions varied in many respects, the state of the weather probably being an important factor. The E.M.F. between the rails was on one occasion as much as 28 millivolts, and differences of 8 and 15 millivolts were frequent. It appears probable to the authors that the effects are due to earth currents, and they suggest that by making the galvanometer very sensitive its deflections might furnish a means of studying their variation.

Dr. C. CHREE expressed his interest in the paper, and remarked that it was often more desirable to avoid earth currents than to record them. He questioned whether the effects described by the authors were due to earth currents, and asked if the authors had satisfied themselves that the variations in the galvanometer deflections were not due to magnetic storms.

Dr. J. A. HARKER described some experiments similar to those made by the authors. In his case, wires from a galvanometer were connected to two large plates about 200 yards apart and about 5 feet underground. He found that the effects varied with the weather.

INSTITUTE OF CHEMISTRY.

Annual General Meeting, March 1st, 1906.

Mr. DAVID HOWARD, President, in the Chair.

THE Accounts for 1905 having been submitted by the Hon. Treasurer, Mr. A. GORDON SALAMON, and duly received, Dr. JOHN A. VOELCKER moved the adoption of the Annual Report. He congratulated the Council on their work during the past year, and he specially alluded to the steps they had taken to protect the interests of professional chemists. Mr. ARTHUR E. EKINS seconded, and the Report was formally received and adopted.

Scutineers having been appointed to count the votes sent in for the election of the Officers and Members of Council, and ballot having been taken for the election of

Censors, the Meeting proceeded to select the Honorary Auditors, and Messrs. Robert E. Alison, W. T. Burgess, and P. A. E. Richards were appointed.

The PRESIDENT then delivered his Address. After referring to the progress made by the Institute during his term of office as President, he dealt more particularly with the history of the third and last year.

While the roll of Fellows and Associates has steadily increased, he noted with regret the death of some of the original Fellows, and he mentioned especially John Lloyd Bullock, largely to whose incentive was due the foundation of the Royal College of Chemistry and the invitation to Hofmann to come to London. How much that meant to not a few, including several Past-Presidents of the Institute, it would take long to tell.

The Institute was examining over 100 candidates yearly, and arrangements would be made from time to time for examinations in the Colonies.

Mr. Howard alluded to the improvement in the financial position of the Institute and to the steady growth of the library. He paid a high tribute to the services of Prof. Adrian J. Brown, who has held the appointment of Examiner to the Institute in Biological Chemistry since 1901, and stated that, as his term of office had expired, the Council had selected Dr. Arthur Harden, of the Lister Institute, as his successor.

He also referred to the new examinations in Technical Chemistry, the first of which will be held in October next. In this connection he expressed the hope that the Institute would in future have for its presidents industrial chemists as well as those eminent for educational and strictly professional work. The chief function of the Institute was to register competent consulting analytical and technical chemists, and the Institute might safely claim to represent the profession. The Council regarded it as their duty to advance the interests of the profession, and, as far as they were able, to maintain it on a sound and satisfactory basis. They had lately been obliged to make representations to authorities whose actions appeared to be detrimental to the profession.

With reference to the gratuitous performance of analyses at Agricultural Colleges, he mentioned that the Board of Agriculture had endeavoured to show that the performance of cheap milk tests had been arranged for educational purposes. At the same time, the Board had stated that these tests were not seriously to be compared with the analyses made by Public Analysts and district analysts. The Council had to complain of more than the milk tests—the Colleges were undertaking all kinds of analyses at nominal fees; of soils, for instance, at half-a-crown. This was undoubtedly injurious to the profession, but the Board in its journal stated that "the demand on the part of land-owners for expert advice from the lecturers had been considerably in excess of what might reasonably have been anticipated." In this matter, the Council had not received a satisfactory response.

He referred to the great advances in chemistry that had been due to the work of private practitioners, giving his opinion that any action which tends to interfere with the individual practitioners would be fatal to progress.

It was with reluctance that the Council had to take up an attitude which might seem in any way antagonistic to the National Physical Laboratory, but they had been obliged to direct the attention of the Executive Committee to the fact that their test pamphlet indicated that they might undertake work which they were forbidden to undertake under the Treasury Report on which the Laboratory was founded. The Executive Committee has recently given their assurance of their desire to avoid any cause for complaint. He was sure that the Council would be glad to see the Laboratory placed on such a sound footing financially that its authorities would have no temptation to extend the work of the Laboratory beyond its proper sphere.

Mr. Howard thought the profession had reached a

somewhat critical stage in its history. With greater facilities for training, and, consequently, a larger supply of chemists, it was evident that only the most efficient could hope to be successful. He believed the demand for chemists was increasing, but authorities and manufacturers were learning that they must have efficiency.

After dealing briefly with the present position of the industrial chemist, and the official professional chemist, he referred to the professors and teachers of chemistry. He objected to the practice, which had grown of late, of blaming the Universities for the loss of certain industries. He maintained that, while chemists were improving so greatly in efficiency, it was absurd to blame the professors. Every decade does not bring a Hofmann, but there were many teachers at the present day under whom the bulk of Fellows and Associates were proud to say they had been trained.

The Institute afforded a great benefit to the public by aiding its discrimination in the selection of competent chemists of acknowledged ability and professional integrity; they could rely on each Fellow and Associate to further the reputation of the Institute by his character and conduct, the soundness of his work, and by the cultivation of professional feeling.

Mr. Howard then referred to the new President, Prof. Percy F. Frankland, who had long been associated with the Institute, and whose father, Sir Edward Frankland, was the founder and first President. He concluded by saying how much he had appreciated his position as President of the Institute, and he spoke in warm terms of the co-operation of the Councils with whom he had worked.

A vote of thanks for the Address was moved by Mr. THOS. TYRER, seconded by Prof. J. MILLAR THOMSON, supported by Mr. R. J. FRISWELL, and carried unanimously.

On the report of the Scrutineers the result of the balloting for the selection of Censors was submitted, and Mr. David Howard, Sir William Ramsay, K.C.B., F.R.S., Sir Thos. Stevenson, M.D., and Prof. J. Millar Thomson, F.R.S., were declared elected.

The Officers and Members of Council for the ensuing year were then declared elected as follows:—

President—Percy Faraday Frankland, LL.D., Ph.D., F.R.S.

Vice-Presidents—Edward John Bevan; Edward Divers, M.D., D.Sc., F.R.S.; David Howard; Edmund Albert Letts, D.Sc.; Edmund James Mills, D.Sc., F.R.S.; Sir William Ramsay, K.C.B., LL.D., F.R.S.

Hon. Treasurer—Alfred Gordon Salamon, A.R.S.M.

Members of Council—Adrian John Brown, M.Sc.; Lt.-Col. Charles Edward Cassal; Arthur Crozier Claudet, A.R.S.M.; John Norman Collie, Ph.D., F.R.S.; James Kear Colwell; Cecil Howard Cribb, B.Sc.; Henry John Horstman Fenton, M.A., F.R.S.; Martin Onslow Forster, D.Sc., F.R.S.; Richard John Friswell; William Gowland, A.R.S.M.; Arthur George Green; Henry George Greenish; Oscar Guttman; James Hendrick, B.Sc.; Egbert Grant Hooper; Herbert Jackson; Arthur Robert Ling; Henry de Mosenthal; Henry Droop Richmond; Alfred Smetham; Arthur Smithells, B.Sc., F.R.S.; John Edward Stead, F.R.S.; David Alexander Sutherland; Francis Napier Sutton; Edward William Voelcker, A.R.S.M.; William Palmer Wynne, D.Sc., F.R.S.; Sydney Young, D.Sc., F.R.S.

On the motion of Lt.-Col. CHARLES E. CASSAL, seconded by Mr. P. GERALD SANFORD, a vote of thanks was accorded to the retiring Officers and Members of Council. The PRESIDENT having replied, the meeting terminated.

Royal Institution.—A General Monthly Meeting of the Members of the Royal Institution was held on the 5th inst., The Duke of Northumberland, K.G., President, in the Chair. Messrs. Frank Green, A. W. Oke, N. M. Ogle, H. F. Pooley, H. Taverner, and A. B. Thomas were elected Members.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances de l'Académie des Sciences. Vol. cxlii., No. 6, February 5, 1906.

Insoluble Potassium Compounds contained in the Trunk and Bark of Oak Trees.—M. Berthelot.—The author makes a series of experiments on the potassium salts found in the trunk and bark of oak trees. He finds that insoluble salts exist in very much smaller proportion than in the leaves, this being apparently due to the fact that the leaves are the terminus of the ascendant circulation of the liquids, and so receive an accumulation of insoluble matter.

Rotatory Power of the Hexahydrobenzylidene- and Cēnanthylidene- camphors and their Corresponding Saturated Derivatives.—A. Haller and F. March.—The authors' experiments prove—(1) that the specific rotatory power of hexahydrobenzylidene and cēnanthylidene camphors are considerably less than the rotatory power of the corresponding benzenic compounds; (2) that the rotation of the saturated acyl-derivatives is inferior to that of the corresponding non-saturated derivatives; (3) that the nature of the saturated lateral chains C_6H_{11} , C_6H_5 , whether they are cyclic or aliphatic, does not seem to sensibly modify the rotatory power in the two series respectively. The author therefore concludes that in benzylidene camphor and its analogues, in the same way as in benzyl camphors, it is the non-saturated character of the benzenic radical which exercises an action on the elevation of the rotatory power of the asymmetric molecule to which this radical is attached.

Condensation of Acetylenic Nitriles with Alcohols. General Method of Synthesis of β -Substituted β -Oxalcoyl Acrylic Nitriles.—Ch. Moureu and I. Lazennec.—When an acetylenic nitrile, $R-C\equiv C-CN$, is put in contact with a solution of methylate or ethylate of sodium in the corresponding alcohol, a brisk reaction takes place. After heating for two or three hours and then cooling, an almost colourless oil is precipitated, which consists of a mixture in varying proportions of β -oxalcoyl ethylenic nitrile and β -acetalic nitrile.

Attempted Reduction in the Diphenylmethane Series.—H. Duval.—In a previous research the author described the method of preparation of azoxy- and azodiamino diphenylmethane, and he now describes his experiments on the influence of reduction and benzidination on these compounds.

Cyclohexylacetone.—P. Freundler.—For some time the author has made various unsuccessful attempts to prepare cyclohexylacetone, $C_6H_{11}.CH_2.CO.CH_3$. He now succeeds in obtaining this ketone, but with a poor yield. The various methods of preparation are described, the most successful being the condensation of hexahydrobenzyl magnesium iodide with acetic aldehyde, and oxidising the secondary alcohol thus obtained with the chromic mixture:—

$$C_6H_5.CH_2.MgI + CHO.CH_3 = C_6H_5.CH_2.CH \begin{smallmatrix} OMgI \\ CH_3 \end{smallmatrix}$$

$$C_6H_5.CH_2.CHOH.CH_3 + O = H_2O + C_6H_5.CH_2.CO.CH_3.$$

The secondary alcohol, of which the yield is about 40 per cent, is accompanied by superior products.

Berichte der Deutschen Chemischen Gesellschaft.
Vol. xxxviii., No. 1, 1906.

Arsenic Pentafluoride.—Otto Ruff and Hugo Graf.—Arsenic pentafluoride is best prepared in a platinum retort connected with an ascending platinum condenser, which leads to a platinum receiver, although glass vessels may be

substituted; they must, however, be carefully dried before use, and no rubber corks, &c., must be employed, because the presence of the least trace of organic matter leads to the formation of hydrofluoric acid. In the retort 0.2 mol. of cooled antimony pentafluoride, and 0.1 mol. of cooled arsenic trifluoride are placed, all moisture being carefully excluded. Then the mixture is cooled to -20° and $\frac{1}{2}$ mol. of cooled bromine is added. While the receiver is still cooled by means of liquid air, the retort is allowed gradually to acquire the temperature of the surroundings, and is finally heated to 55° for about half-an-hour on a water-bath. At the end of this time about the theoretical quantity of arsenic pentafluoride will be found in the receiver mixed with some bromine. To separate this latter, the mixture must be fractionated. For this purpose the receiver is removed, and connected with a similar receiver which has previously been very carefully dried, and the first receiver is taken out of the liquid air, while the second is cooled by the same agent. The distillation of the pentafluoride begins at once, and is finished when the vessel has acquired the temperature of the surroundings, nearly all the bromine remaining behind in the first vessel. To complete the separation, the distillation must be repeated. The reaction which occurs is represented by the equation: $2SbF_5 + AsF_3 + Br_2 = 2SbF_4Br + AsF_5$. The authors have not investigated whether the antimony bromine fluoride is a simple compound or a mixture. Arsenic pentafluoride is a colourless gas, which condenses to a clear slightly yellowish liquid at -53° , and solidifies at -80° to a white mass. The gas dissolves in water and alkali solutions, much heat being evolved. Its great affinity for water is shown by the dense white clouds which are formed when it comes in contact with the air. It does not attack perfectly dry glass in the cold, but a violent reaction occurs if the least trace of moisture or of hydrofluoric acid is present. On heating, even dry glass reacts with the gas, silicon tetrafluoride being formed and a white volatile substance being separated, obviously consisting of arsenic pentoxide. Silicon does not react in the cold, but on heating, arsenic is separated and silicon tetrafluoride formed. Sulphur and arsenic pentafluoride react upon one another very slowly in the cold, the sulphur becoming first orange and then black, and finally a sticky mass is formed. This smells strongly of sulphur monochloride or bromide, but soon loses the smell in air, the black product simultaneously turning yellow and crystallising. Dry phosphorus is only slightly attacked, even on warming. Iodine reacts readily in the cold. Copper is not attacked in the cold, but blackens on warming. Zinc, iron, and bismuth darken on heating in the gas, obviously in consequence of the separation of arsenic and formation of fluorides. Lead and mercury react particularly energetically on heating. Tungsten is not affected. The pentafluoride is soluble in liquid arsenic trifluoride, heat being evolved to a slight extent. Generally a vigorous reaction occurs with organic substances. Dry paper and sugar are not altered by the gas, but the moist substances are rapidly charred. Paraffin and wax are somewhat more resistant, but are gradually blackened. The density corresponds approximately to the molecular weight represented by the formula AsF_5 .

Acceleration of certain Oxidation Reactions by Prussic Acid.—A. S. Loevenhart.—The author has come to the conclusion that the catalytic decomposition of hydrogen peroxide into water and molecular oxygen, and the indirect oxidations by means of hydrogen peroxide, are phenomena which are closely allied to one another. The grounds for this belief are:—(1) All substances so far investigated which catalyse hydrogen peroxide also give rise to indirect oxidations by means of hydrogen peroxide; (2) the two processes are equally influenced by temperature; (3) substances which retard one of these processes have the same effect on the other; e.g., prussic acid retards the catalysis of hydrogen peroxide by platinum black, and also the oxidations produced by means of hydrogen peroxide. This theory is confirmed by the investigation

of the oxidation of formic acid by means of hydrogen peroxide in presence of copper sulphate, which is accelerated by the presence of prussic acid. Apparently the more stable cupric compounds can be converted into cuprous compounds only by very strong reducing agents, while cupric cyanide is very rapidly reduced to the cuprous salt, and thus the acceleration is due to the formation of the cyanogen compound with copper. It is evident that prussic acid cannot be regarded as an anti-catalyser, and indeed it is, generally speaking, not possible to say definitely that any given substance is an anti-catalyser, for the same substance which accelerates the action of one catalyser may retard that of another.

Titrimetric Method for the Quantitative Determination of Pentoses.—Adolph Jolles.—The pentoses, or substances yielding them, are first converted into furfural by distillation with hydrochloric acid, and the furfural is driven over by a current of steam. An aliquot part of the distillate is neutralised and a measured quantity of bisulphite added, when the following reaction occurs:— $C_4H_5O.CHO + KHSO_3 = C_4H_5O.CH(OH).SO_3K$. Thus each molecule of furfural requires one molecule of bisulphite. After two hours the excess of bisulphite is titrated back with iodine solution; each c.c. of normal bisulphite corresponds to 75.05 m.grms. of pentose. The same method can be used for the determination of furfural.

Cyanuric Acid as Pseudo Acid.—A. Hantzsch.—Cyanuric acid dissolves to a clear solution in fairly concentrated caustic soda at the ordinary temperature, but from this solution, which does not appear supersaturated, and contains only secondary sodium salt, on heating tertiary salt is deposited, which is not dissolved by the mother-liquor at the ordinary temperature. The formula of the secondary salt is $[C(ONa):N]_2[CO.NH]$, and that of the tertiary $[C(ONa):N]_3$, and at the higher temperature the last group, $CO.NH$, of the secondary salt isomerises to $C(OH):N$, and is fixed as $C(ONa):N$. By the conductivity it may be shown that in an aqueous solution of trisodium salt at the ordinary temperature, one molecule of free soda is present. After evaporation of the solution at the ordinary temperature a mixture of disodium salt and free soda remains behind. This phenomenon may be regarded as an hydrolysis of the cyanurate, abnormally varying with the temperature. For the salts $C_3N_3O_3Me_3$ are at ordinary temperatures totally hydrolysed to $C_3N_3O_3Na_2H + NaOH$, but while with increasing temperature in normal cases the hydrolysis, on account of the greatly increasing dissociation of water, also increases; the opposite occurs here in consequence of the intramolecular transposition, $CO.NH + NaOH \rightarrow C(ONa):N + H_2O$. A decrease of the hydrolysis of alkali salts as the temperature increases is thus a new criterion of an intramolecular transposition, or, in other words, a criterion whether the salts in question have been formed from a pseudo acid. This test is naturally applicable only in the case of those pseudo acids which are transposed into such feeble true acids that their alkali salts suffer appreciable hydrolysis.

Preparation of Trialkyl Stilbine, Arsine, and Phosphine by Grignard's Reaction.—Harold Hibbert.—Auger and Billy have found that by the action of magnesium ethyl bromide upon excess of phosphorus, arsenic, or antimony trichloride, no trialkyl compounds are formed, or only very small amounts. But if the trichlorides are allowed to react with three or more molecules of the magnesium alkyl compound in ethereal solution, a yield of up to 75 per cent of trialkyl derivative is obtained. This can then be distilled in a current of carbon dioxide. In the case of the phosphorus derivative the yield is very small unless a very large excess of magnesium ethyl bromide is used. The greater part of the triethyl phosphine does not distil over till a temperature of 160–200° is reached, which seems to show that it is not formed directly, as in the case of the arsenic or antimony compound, but that either brominated by-products or addition products result, and give up free triethyl phosphine

at the higher temperature. The yield of phosphine amounts to 70 per cent of the theory.

Action of Oxygen on Aliphatic Amines in presence of Copper.—Wilhelm Traube and Albert Schönewald.—In the presence of oxygen aqueous solutions of the primary amines, e.g., ethylamine, rapidly attack copper, and the base is oxidised, the alkyl group being converted into aldehyde, while the nitrogen splits off as ammonia. No nitrites could be detected either as final or by-products. The proportion of oxygen used for the oxidation of the copper and the formation of aldehyde was 1:1, half the oxygen being combined with the metal, while the other half attacks the amine. With methylamine formaldehyde is similarly obtained and free ammonia, no nitrous acid being formed.

Use of Hydrogen Peroxide for the Quantitative Separation of the Halogens.—P. Jannasch and Fr. Zimmermann.—The mixture of halogens is dissolved in water in a distillation flask, pure glacial acetic acid and hydrogen peroxide are added, and the mixture is then distilled in a current of steam. The iodine is driven over quantitatively in not more than twenty to twenty-five minutes, the liquid being completely decolorised in from three to five minutes. The iodine is received in a vessel containing water, uncrystallised hydrazine sulphate, and the strongest ammonia. After the distillation has been stopped the contents of this vessel are washed into a beaker, acidified with concentrated nitric acid free from chlorine, and precipitated with excess of silver nitrate solution in the cold, the resulting silver iodide being heated until it can be filtered readily. The bromine may be separated from the chlorine in the residue by the addition of large quantities of sulphuric acid, but the authors have not yet finished their investigation of this point.

Properties of Electrolytic Calcium.—L. Doermer.—The author found when experimenting with electrolytic calcium from the Bitterfeld Electrochemical Works that somewhat violent explosions occurred when he struck the metal sharply with a hammer on an anvil. The explosiveness is very much reduced, or is even removed altogether if implements are used which are perfectly free from rust and iron oxide. Working with a rusty anvil explosions were always obtained, while if hammer and anvil had been thoroughly rubbed with emery paper they seldom occurred. On gently warming the metal, before a red heat was reached, hydrogen was set free, and if the metal was then heated to a faint red heat all the gas was rapidly absorbed again. When the electrolytically prepared metal is ignited a glow passes through the mass, which fuses to a doughy melt, consisting of metallic calcium. A flame held over the melt during this process is coloured distinctly yellowish red by volatile calcium compounds. If this process is carried out in a crucible and the doughy mass is kneaded with an iron spatulum, gas flames appear and a calcium regulus is obtained, which after cooling is much more stable towards moist air than ordinary calcium. A piece of this calcium after lying in air for eight days showed no external change, while with ordinary calcium a large amount of hydrate was formed. The same behaviour was noticed towards water, and the unfused calcium is harder, more brittle, and of a yellower colour.

Preparation of Titanium Tetrachloride and Tin Tetrachloride.—Carl Renz.—By the action of carbon tetrachloride and heat upon certain oxides the following reactions occur:— $CCl_4 + MeO = MeCl_2 + COCl_2$, $CCl_4 + 2MeO = 2MeCl_2 + CO_2$. Thus when carbon tetrachloride is led over heated titanium dioxide, carbon dioxide or phosgene and titanium tetrachloride are formed. If chloroform is substituted for carbon tetrachloride this method is excellently suited for the preparation of titanium tetrachloride. The chief reaction proceeds as follows:— $TiO_2 + 2CHCl_3 = TiCl_4 + 2CO + 2HCl$, but according to the temperature various by-reactions also occur. Sometimes a colourless gas, presumably titanium hydride, TiH_4 , results, carbon dioxide and phosgene have also been

detected, and hexachlorbenzene. If stannic oxide is substituted for titanium dioxide, the corresponding tin tetrachloride is formed, but silicon tetrachloride cannot be prepared by the same method.

MEETINGS FOR THE WEEK.

- MONDAY, 12th.**—Society of Arts, 8. (Cantor Lectures). "Fire, Fire Risks, and Fire Extinction," by Vivian B. Lewes.
- TUESDAY, 13th.**—Royal Institution, 5. "Food and Nutrition," by Prof. William Stirling, M.D., &c.
- WEDNESDAY, 14th.**—Society of Arts, 8. "Imperial Organisation from a Business Point of View," by Geoffrey Drage.
- THURSDAY, 15th.**—Royal Institution, 5. "The Physiology of Plants," by Francis Darwin, M.A., &c.
- Society of Arts, 4.30. "The Languages of India and the Linguistic Survey," by Dr. George A. Grierson, C.I.E.
- Chemical, 8.30. "Interaction of well-dried Mixtures of Hydrocarbons and Oxygen," by W. A. Bone and G. W. Andrew. "Explosive Combustion of Hydrocarbons," by W. A. Bone and J. Drugman. "The occurrence of Marsh Gas amongst the Decomposition Products of certain Nitrogenous Bases as a Source of Error in the Determination of Nitrogen by the Absolute Method," by P. Haas. "Studies on Comparative Cryoscopy—Part IV., The Hydrocarbons and their Halogen Derivatives in Phenol Solution," by P. W. Robertson. "Displacement of Acid Radicles—I., Displacement of the Chloride and Nitrate Radicles," by A. F. Joseph.
- FRIDAY, 16th.**—Royal Institution, 9. "How to Improve Telephony," by W. Duddell.
- SATURDAY, 17th.**—Royal Institution, 3. "The Corpuscular Theory of Matter," by Prof. J. J. Thomson, F.R.S., &c.

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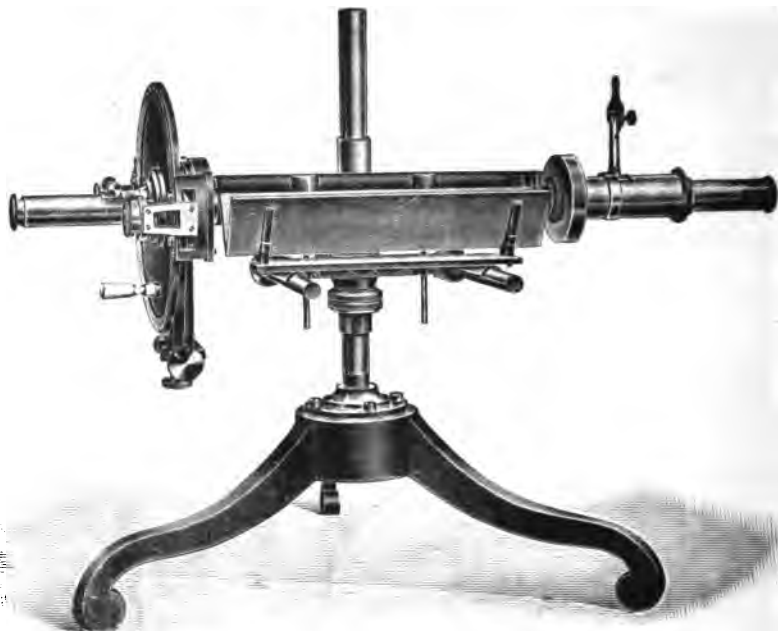
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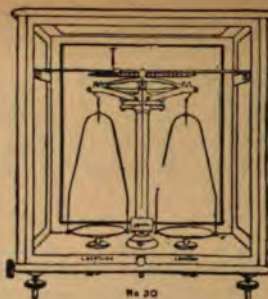
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THE CHEMICAL NEWS.

VOL. XCIII., No. 2416.

REDUCTION OF SULPHURIC ACID AND OTHER SUBSTANCES.

By Dr. T. L. PHIPSON.

WHEN a piece of metallic sodium is allowed to fall into monohydrated sulphuric acid, sp. gr. 1.845, contained in a small porcelain capsule, the reaction is extremely violent, accompanied by a loud hissing sound, great heat, and copious projections of vapour. The chief product remaining in the porcelain capsule is a yellow sulphide of sodium.

A good many years have elapsed since I made this dangerous experiment, which requires the greatest precautions on account of the projections, and I never published it. Soon after I used metallic magnesium in my experiments of this kind (see my note on "Magnesium" in the *Proceedings of the Royal Society*). Thus I was the first to obtain, by the aid of a simple spirit-lamp, carbon from carbonic acid, boron from boric acid, silicon from silica, and zirconium from zirconia.

The carbon thus obtained from pure anhydrous carbonate of soda in a thin porcelain crucible, well closed, has not the aspect of graphite, nor anthracite, like that in the beautiful little quartz crystals from the mountain limestone at Maffes, in Belgium; but when the magnesia and soda that are present are washed out by a little weak hydrochloric acid, this carbon has the dull black appearance of pulverised wood-charcoal. A peculiar emission of light seen through the thin sides of the porcelain crucible announces when the reduction has taken place.

The silicon thus produced has a brownish colour, the boron also, but the zirconium has a peculiar velvet-black aspect.

M. Moissan stated a short time ago in the *Comptes Rendus* that this process of mine is the only one that yields absolutely pure zirconium.

THE NEUTRAL HYDROCHLORATE OF QUININE.

By H. CARETTE.

In a former paper (*Journ. de Pharm. et de Chim.*, [6], vol. xx., p. 347), on the neutral hydrochlorate of quinine, it was shown that this salt can be crystallised with alcohol of crystallisation. Shortly afterwards I learnt through the same Journal ([6], vol. xx., p. 550), that M. Biscaro had some years previously (*Boll. Farm.*, 1901, [4], vol. xl., p. 105), observed the presence of alcohol in certain crystals of the same salt. We both found losses identical in amount, on desiccating the salt crystallised in alcohol; viz., 14.33 to 14.34 per cent; but while I interpreted this result of the analyses by the loss of 1½ molecules of alcohol, or 14.80 per cent, M. Biscaro attributed the loss observed to the simultaneous presence in the salt of a molecule of alcohol and a molecule of water, that is to say 13.88 per cent. In view of this divergence in the interpretation of identical analytical results, I thought it advisable to repeat the examination of the crystallisation of the neutral hydrochlorate of quinine. I now give the results of my fresh experiments.

One result of my later experiments is that by the crystallisation in alcohol absolutely free from water, the neutral hydrochlorate of quinine furnishes crystals which had not previously been observed either by M. Biscaro or

myself. These crystals were in the form of white nodules containing only one molecule of alcohol of crystallisation. The neutral hydrochlorate of quinine, when crystallised in alcohol at 95 per cent, fixes simultaneously one molecule of alcohol and one molecule of water, as shown by M. Biscaro.

I have made a comparative examination of the products of the crystallisation of the neutral hydrochlorate of quinine in alcohol completely free from water, and in alcohol containing small quantities of water.

I first of all used alcohol completely dried by means of anhydrous baryta, preventing in all the operations any contact with moist air which might hydrate the alcohol.

The neutral hydrochlorate of quinine dried by means of a sufficiently prolonged exposure in an oven at 95°, was then dissolved in the hot absolute alcohol. On crystallising this alcoholic liquor by cooling, the salt is deposited as white opaque nodules, composed of fine needles.

In the oven at 95°, 0.65 gm. of this salt, well drained on filter paper, lost 0.0745 gm., or 11.40 per cent in weight, the formula $C_{20}H_{24}N_2O_2 \cdot 2HCl + C_2H_6O$ requiring 10.38 per cent.

These mamellated crystals thus contained 1 molecule of alcohol. They have somewhat the appearance of those obtained from an aqueous solution, but which contained 2½ molecules of water. They have not, however, been observed previously. When exposed to the air they rapidly lose their alcohol of crystallisation, but at the same time take up moisture from the atmosphere; thus they change into the neutral hydrochlorate of quinine with 2½ molecules of water. Even in a bottle imperfectly closed with a cork they lose their alcohol fairly rapidly.

In a second series of experiments I crystallised the neutral hydrochlorate of quinine in ordinary absolute alcohol, that is to say in alcohol containing slight traces of water. I dissolved the hydrochlorate with heat in this so-called absolute alcohol without taking the precautions pointed out in the preceding experiments, that is to say without preventing contact with moist air.

The crystals deposited, after starting them by introducing a crystal containing 1 molecule of alcohol and 1 molecule of water of crystallisation, are similar to those described previously. After desiccation they gave the same loss already shown by M. Biscaro and myself.

I wished to make certain whether the alcohol they lose under the action of heat contains water; I therefore distilled them on a bath heated to 150° and collected the liquid in a tube containing anhydrous colourless sulphate of copper. The salt became blue on contact, thus showing the presence of a certain proportion of water. I further observed that the distillate contained alcohol; it was combustible in the air.

Thus it is established that the traces of water contained in ordinary absolute alcohol, and those that this same alcohol may take up from the air, are combined in the crystals of the neutral hydrochlorate of quinine deposited in the solution. The crystals thus obtained, dried at 37° in a dry atmosphere, lose one molecule of alcohol and half a molecule of water. They form a salt with half a molecule of water, which is stable in dry air. In moist air this salt takes up water, forming the salt with two and a-half molecules of water.

I have also effected the crystallisation of the neutral chlorohydrate of quinine by adding directly a very small quantity of water to the absolute alcohol used as a solvent. I first obtained the crystals containing one molecule of alcohol; then after starting the crystallisation of the mixture with a crystal containing one molecule of alcohol and one molecule of water, crystals containing the latter amounts of alcohol and water were deposited on the first-named.

This experiment leaves no doubt that we have here to deal with two different crystals.

The following is no less conclusive:—When to a crystallisation of the salt containing one molecule of alcohol, immersed in its own mother-liquor of absolute alcohol, we

add a very small quantity of water, and start the action by means of a crystal containing one molecule of alcohol and one molecule of water, the crystals containing one molecule of alcohol only gradually change into crystals containing one molecule of alcohol and one molecule of water; this is also easily observed by their change of appearance. When the proportion of alcohol to water is appropriate we are able to obtain both kinds of crystals simultaneously and directly.

I may add that the crystals containing one molecule of alcohol, when exposed to slightly moist air in a confined space, change their appearance; they become transparent rapidly and take all the characteristics of the crystals containing one molecule of alcohol and one molecule of water.—*Journ. de Pharm. et de Chim.*, Series 6, vol. xxii., No. 7.

THE DECOMPOSITION OF SULPHATE OF AMMONIUM BY HOT SULPHURIC ACID IN THE PRESENCE OF PLATINUM.

By MARCEL DELÉPINE.

I HAVE observed that the intervention of spongy platinum to regulate the boiling of sulphuric acid, as is done in the estimations of nitrogen by Kjeldahl's method, causes a heavy or the total loss of ammonia, and it appeared to me to be of interest to find out definitely the mechanism of this reaction, which has some connection with my old experiments on the insufficiency of Kjeldahl's method for the estimation of nitrogen in chloroplatinates (Delépine, *Bull. Soc. Chim.*, 1895, Series 3, xiii., p. 222).

These experiments were confirmed by W. van Dam in a more extended research (*Rev. Trav. Chim. P.-B.*, 1895, xiv., p. 217); this writer has further accepted my opinion put forward as a hypothesis, that the chloroplatinates brought about their own decomposition by a sort of internal dehydrogenation as follows:— $\text{PtCl}_6(\text{NH}_4)_2 + \text{Cl}_6 = \text{N}_2 + \text{PtCl}_4 + 8\text{HCl}$, the chlorine being furnished by the decomposition of the chloride of platinum; but without verifying it, he still imagined that the platinum-black deposited during the reaction played an active rôle; this supposition was based primarily on the fact that the chloraurates did not cause any loss of nitrogen, although chloride of gold is as easily decomposed as chloride of platinum.

I would here mention that if we refer to the numerous estimations of nitrogen made by M. van Dam in various chloroplatinates with organic bases, we shall see that the addition of sulphate of potassium, according to Gunning's modification, causes greater losses of nitrogen than if we use sulphuric acid only; these losses may be even total. They are also very high if we have to heat for a long time in order to destroy the organic matter.

All these observations are fully explained by my new experiments.

If we boil sulphuric acid containing sulphate of ammonium, in the presence of spongy platinum or platinum-foil, we notice a loss of nitrogen which becomes greater as the experiment is prolonged, and, for the same time, that it takes place at a higher temperature; this is easy to regulate between 338° and 370° by adding from 0 to 50 per cent of sulphate of potassium to the sulphuric acid. If the amount of sulphate of ammonium is sufficient the platinum does not change appreciably in weight; the reaction thus has the character of those reactions called *catalytic*.

The nitrogen disappears in the form of gas (a fact already observed by M. van Dam). At the same time sulphurous acid gas is formed; thus it is only natural to think that the hydrogen of the ammonia is burnt by a portion of the oxygen of the sulphuric acid, according to the equation $2\text{SO}_4\text{H}(\text{NH}_4) + \text{SO}_4\text{H}_2$ or $\text{SO}_4(\text{NH}_4)_2 + 2\text{SO}_4\text{H}_2 = \text{N}_2 + 3\text{SO}_2 + 6\text{H}_2\text{O}$.

This equation was verified by a double series of experi-

ments. We introduced into sulphuric acid (both with and without SO_4K_2) in the presence of platinum, a known quantity of sulphate of ammonium; after the reaction we estimated the loss of nitrogen by difference while estimating the remaining ammonia. On the other hand, thanks to an arrangement easily fitted up, we were able either to collect the nitrogen directly, or to direct the sulphurous acid gas formed into a titrated solution of iodine, and by means of the above equation to calculate the loss of nitrogen corresponding to this gas. The results are very concordant, as is shown by the following figures:—

N from $\text{SO}_4(\text{NH}_4)_2$.	N lost according to the remaining ammonia.	Nitrogen collected.
0.0683	0.0287	0.0274
0.0713	0.0189	0.0193
0.0715	0.0340	0.0342
0.0431	0.0174	0.0179
0.0716	0.0559	0.0539
0.0711	0.0046	0.0044
0.0712	0.0102	0.0102
0.0681	0.0154	0.0160
0.0672	0.0258	0.0263

To make the speed of this destruction more clear, I will point out that in one hour 0.03 grm. of spongy platinum, from the calcination of chloroplatinate of aniline, caused a loss of 0.008 grm. of nitrogen in a mixture formed of 30 c.c. SO_4H_2 , 20 grms. SO_4K_2 , and 0.30 grm. $\text{SO}_4(\text{NH}_4)_2$; without the addition of potassic sulphate the loss is five or ten times less during the same time.

We may attempt to account for the above phenomena in two ways:—

1. Hot sulphuric acid in contact with platinum splits up into $\text{O} + \text{H}_2\text{O} + \text{SO}_2$, as at a higher temperature; the oxygen is not given off but combines with the hydrogen of the ammonia.

2. Sulphuric acid attacks platinum and forms a sulphate, which is destroyed by the ammoniacal salt, regenerating the platinum:— $4\text{SO}_4\text{H}_2 + \text{Pt} = (\text{SO}_4)_2\text{Pt} + 2\text{SO}_2 + 4\text{H}_2\text{O}$, $3(\text{SO}_4)_2\text{Pt} + 2\text{SO}_4(\text{NH}_4)_2 = 2\text{N}_2 + 3\text{Pt} + 8\text{SO}_4\text{H}_2$.

The second process is the true one.

It can be proved that sulphuric acid attacks platinum almost at the speed it does silver; the coloured solution obtained deposits the greater portion of its platinum if we heat with sulphate of ammonium. We have, further, an indirect proof, in that spongy gold and iridium, which are not attacked by sulphuric acid, do not cause any loss of nitrogen if they are substituted for the platinum.

To sum up, platinum brings about the decomposition of sulphate of ammonium by boiling sulphuric acid, and should never be used in working Kjeldahl's method.

This decomposition takes place thanks to the momentary solution of the platinum. The attack of the platinum by sulphuric acid having been regarded as a controversial matter, or having been diversely interpreted, I have examined it specially in this paper.—*Bull. Soc. Chim.*, Series 3, vol. xxxv., No. 1.

Molecular Weight of Silver Vapour.—H. v. Wartenberg.—Using the apparatus devised by Nernst for determining vapour densities, and a modification of Victor Meyer's method, the author has determined the vapour density of silver. In nine experiments he found molecular weights varying from 107 to 147, thus showing that the vapour is monatomic in the neighbourhood of the boiling-point.—*Berichte*, xxxviii., No. 2.

* The opinion that boiling sulphuric acid splits up on contact with platinum into $\text{O} + \text{H}_2\text{O} + \text{SO}_2$ was offered as an explanation of this experiment by Redwood (*Pharm. Journ.*, [2], v., p. 601) that the distillation of sulphuric acid in a platinum retort gives a slightly weakened acid ($d = 1.842$ instead of 1.843), and with a sulphurous odour. Now, as Redwood added sulphate of ammonium before distilling, his observation bears on the facts I have observed, and which I interpret in Paragraph 2. When operating in glass retorts Redwood did not observe this phenomenon.

WET AND CRUCIBLE-FIRE METHODS FOR THE ASSAY OF GOLD TELLURIDE ORES, WITH NOTES ON THE ERRORS OCCURRING IN THE OPERATIONS OF FIRE ASSAY AND PARTING.*

By W. F. HILLEBRAND and E. T. ALLEN.

(Continued from p. 111).

PART II. THE ERRORS IN GOLD ASSAYING.

Cupellation Losses.

Absorption and Volatilisation.

THAT the loss of silver in assaying may be very marked is, of course, well known, particularly that loss which is due to too high heat during cupellation. That gold suffers similar losses, though to a much less degree, has been fairly well established by various workers. "That loss of gold takes place in cupellation if this is conducted at too high a temperature, or if the alloy is cupelled without quartation silver, is an already long known experimental fact" (Bodeman-Kerl's "Assaying," 1880, p. 194, translation by Goodyear). The data relating to gold are, however, not so satisfactory as those with respect to silver, and they are at times uncertain or even conflicting. It is in general said that the cupellation of gold may be carried out at a considerably higher temperature than that of silver, and the assayer is commonly advised to complete the final stage of the operation further back in the muffle than the earlier stages. Some authorities insist on the absence of feather litharge at the end. There seems to be in the literature no adequate appreciation of the very marked effect that slight differences in temperature actually do produce on the yield of gold by cupellation, especially if the ore be rich, nor of the very considerable volatilisation loss that accompanies cupel absorption if the heat is not kept at a minimum. Doubtless these facts are known to individual assayers, but they are not sufficiently emphasised in chemical literature, especially that to which practical assayers in this country usually have access. "Many yet believe that the degree of heat has little or no influence on the result" (Roessler in *Dingler's Polytech. Journ.*, 1872, ccvi., p. 186).

Our own experiments, which follow, were all made with proof gold and silver from the Mint. The results are not therefore necessarily comparable with those obtained by assaying ordinary bullion, where the presence of copper is said to demand a higher temperature, and to influence the gold loss.

First, gold alone was cupelled in order to ascertain primarily the loss with increase of temperature, and, secondarily, how this loss was affected by the amount of lead used. Ordinarily six cupels were set in a row, from front to back of the 12 by 6 inch muffle, so that the front cupel always showed at the end abundant feather litharge, and the rear one was at the back-end of the muffle. The cupellations were made in most cases with an altogether unnecessarily large amount of lead (25 grms.), in order to duplicate as nearly as might be the conditions under which the assays described in Part I. were cupelled. Table VIII. shows these results.

Then quartation mixtures were cupelled and the silver as well as the gold losses determined (Table IX.).

The foregoing tables show most strikingly the effect of increasing temperature on the loss of gold (and silver), and that this loss with pure gold is almost wholly by cupel absorption. A difference of position in the muffle amounting to no more than the width of a cupel is of great moment. The percentages given in the last two columns of Series A, Table VIII., and of Series B, Table IX., are of course subject to considerable probable error because of the difficulty of weighing such small beads to hundredths of a milligram, and the probability of losses

inherent in the operations by which they were obtained. Further, because of the probable retention of traces of lead by the large gold beads (see below, Experiment I.), the volatilisation figures are, if anything, too low and those for absorption too high.

It is clear that the following statements are far from correct:—

"A high temperature has no ill effect, the loss of gold in cupellation being so small that it need not be considered. It is about balanced by the silver left in the gold after parting. The cupellation loss is about 0.07 per cent" (Beringer, "A Text-book of Assaying," 1890, p. 128).

"The loss of gold by cupellation is insignificant (about 0.7 per 1000), compensated, moreover, by the small quantity of silver which the gold retains after parting (Campredon, "Guide Pratique du Chimiste Métallurgiste," 1898, p. 689).

"All the silver lost is absorbed by the cupel" (Campredon, *Ibid.*, p. 304).

Our experience goes to show that, as a general direction, the following should be modified, for with pure gold and telluride ores no higher heat is needed than for silver cupellation.

"A high heat is maintained throughout the operation [cupellation], and especially at the time of brightening; that is to say, a higher degree than is admissible in a silver assay . . ." (Aron, "Assaying," 1900, p. 36).

The average loss named in the first two of these extracts is from three to seven times less than the lowest of those in Tables VIII. and IX.—those suffered at the lowest permissible limit of temperature.

Though not connected with the present discussion, it is impossible to see why the following, credited to Aidarow, should be true:—

"The gold, however, only soaks into the cupel when it has been scorified with lead beforehand. If gold is added to lead that is already fused on the cupel, no loss of gold takes place on cupellation" (Bodeman-Kerl, "Assaying," translated by Goodyear, 1880, p. 171).

That a small but appreciable portion of the total loss is due to volatilisation the results of Series A, Table VIII., and Series B, Table IX., clearly show. That such loss occurs is mentioned by some writers (Makins, *Quart. Journ. Chem. Soc.*, 1861, xiii., p. 97; Roessler in *Dingler's Polytech. Journ.*, 1872, ccvi., p. 189; Ricketts and Miller, "Notes on Assaying," 1897, pp. 128 and 129); but most writers make no mention of it. According to Rose ("Metallurgy of Gold," p. 479), 10 per cent of the total loss in bullion assay is by volatilisation, a proportion roughly agreeing with the returns in Table VIII., Series A, final columns.

That gold volatilises slowly if kept above its melting-point Napier has shown (*Quart. Journ. Chem. Soc.*, 1858, x., p. 229), but this condition does not ordinarily, if at all, obtain after the bead has brightened, so that the direction that it is quite safe to leave gold beads in the muffle after brightening for almost any length of time seemed probably justified. To test the point, however, a number of trials were made by cupelling several beads simultaneously and taking them out at intervals of one-half, five, and twenty minutes after pushing them back in the muffle to brighten. No constant losses were suffered by those left in for the longer periods, but curiously enough there seemed to be a slight tendency to increase in weight. To what this could be attributed is not clear.

On their face the results of Series C, Table VIII., as compared with Series B of the same table, seem to indicate that the amount of lead exerts a very marked effect on the gold loss. Roessler is fully persuaded from his own table of tests that this is generally true (*Dingler's Polytech. Journ.*, 1872, ccvi., p. 188). But compare for a moment the three series of Table IX. The same amount of lead was used in them all (25 grms.), and the absolute losses of gold with 30 m.grms., and with 10 m.grms. were practically identical, while with 15 m.grms. (Series B) they dropped to about one-third. Here the only seemingly probable explanation is that the temperature

* United States Geological Survey. Bulletin No. 253. (Series E, Chemistry and Physics, 44).

TABLE VIII.—*Losses of Gold when Cupelled without Silver as affected by Temperature and Amount of Lead.*
(25 grms. in Series A and B, 5 grms. in Series C).

		Gold taken. M.grms.	Gold found. M.grms.	Loss.		In cupels. M.grms.	Volatilised. M.grm.	Loss by absorption. Per cent.	Loss by volatilisation. Per cent.
				M.grm.	Per cent.				
Series A	1.	30.58	30.47	0.11	0.36	0.15	—	136	—
	2.	30.32	29.96	0.36	1.19	0.29	0.07	80	20
	3.	30.63	30.09	0.54	1.76	0.57	—	105	—
	4.	30.45	29.30	1.15	3.78	0.89	0.26	77	23
	5.	30.16	28.90	1.26	4.17	1.17	0.09	93	7
	6.	30.66	29.30	1.36	4.43	1.25	0.11	92	8
Series B	1.	10.34	10.31	0.03	0.29	—	—	—	—
	2.	10.25	9.77	0.48	4.68	—	—	—	—
	3.	10.29	10.15	0.14	1.36	Litharge formed faster than it could be absorbed during much of the time, yet the furnace ran at an unusually high heat.			
	4.	10.27	9.20	1.07	10.42				
	5.	10.17	8.56	1.67	16.43	—	—	—	—
	6.	Lost	—	—	—	—	—	—	—
Series C	1.	10.36	10.33	0.03	0.29	5 grms. lead.			
	2.	10.31	10.23	0.08	0.78				
	3.	10.31	10.16	0.15	1.45				
	4.	10.17	9.87	0.30	2.95				
	5.	10.29	9.84	0.45	4.37				
	6.	Lost	—	—	—				

TABLE IX.—*Losses of Gold and Silver when Cupelled together.*
(25 grms. Lead).

(25 grms. Lead).																			
Gold.		Gold loss.				Silver.		Silver loss.				In cupels.				Percentage of loss.			
Taken.	Found.					Absorbed.	Volatilised.					Absorbed.	Volatilised.						
M.grms.	M.grms.	M.grms.	Per cent.	M.grms.	M.grms.	M.grms.	Per cent.	Au.	Ag.	Au.	Ag.	Au.	Ag.	Au.	Ag.				
Series A.																			
1.	30.06	29.91	0.15	0.50	90.51	88.97	1.54	1.70											
2.	30.40	30.03	0.37	1.22	90.19	86.82	3.37	3.73											
3.	30.60	29.89	0.71	2.32	90.74	85.71	5.03	5.51											
4.	30.07	28.94	1.13	3.76	90.67	83.73	6.94	7.66											
5.	30.61	29.42	1.19	3.89	90.75	83.51	7.24	7.98											
6.	Lost	—	—	—	—	—	—	—											
Series B.																			
1.	15.56	15.53	0.03	0.19	45.06	44.20	0.86	1.91	0.02	0.70	0.01	0.16	67	81	33	19			
2.	15.14	15.08	0.06	0.40	45.19	43.70	1.49	3.30	0.05	1.17	0.01	0.32	83	78	17	22			
3.	15.15	14.92	0.23	1.52	45.41	43.53	1.88	4.14	0.12	1.19	0.11	0.69	52	63	48	37			
4.	15.44	15.12	0.32	2.07	45.30	42.68	2.62	5.78	0.22	1.63	0.10	0.99	67	62	33	38			
5.	15.52	15.12	0.40	2.59	45.59	42.61	2.98	6.55	0.27	2.09	0.13	0.89	67	70	33	30			
6.	15.39	15.02	0.37	2.40	45.05	42.07	2.98	6.61	0.27	2.13	0.10	0.85	73	71	27	29			
Mean													68	71	32	29			
Series C.																			
1.	10.67	10.62	0.05	0.47	30.33	29.67	0.66	2.17											
2.	10.57	10.40	0.17	1.61	30.64	28.90	1.74	5.68											
3.	10.53	9.99	0.54	5.13	30.42	27.32	3.10	10.19											
4.	10.63	9.50	1.13	10.63	30.52	25.64	4.88	15.99											
5.	10.60	9.28	1.32	12.46	30.38	24.81	5.57	18.34											
6.	10.21	8.93	1.28	12.53	30.44	24.75	5.69	18.69											

throughout the muffle must have been appreciably lower with B than with A and C. Hence it is not improbable that a similar explanation may be the proper one to adopt for the low results of Series C in Table VIII. The cause is certainly not conclusively shown, and it has been made plain that slight differences in temperature produce marked effects.

The final columns in Series B, Table IX., are of interest as showing that with quartation alloys not only is the volatilisation loss of gold greater than when pure gold is cupelled, but also that the ratio of volatilisation loss to absorption loss is about the same for both gold and silver, or, roughly, as 30 to 70. We regret that similar comparisons were not made for the other series. It is hoped

that at some future time opportunity may offer to test this and other points of interest more critically. For the present, however, this is impossible owing to the enforced abandonment of the survey's fire-assay laboratory. It is largely due to this latter fact that the work herein described is rather fragmentary and not altogether satisfying.

(To be continued).

Combination of the Rare Metals of the Cerium Group; their Sulphates in particular.—Camille Matignon.—The author claims priority over M. Otto Brill regarding a number of experiments on certain sulphates of the rare earths.—*Comptes Rendus*, cxlii., No. 7.

CONDENSATION COMPOUNDS OF CAMPHOR-
OXALIC ACID AND AMINES.*

SEVENTH COMMUNICATION ON CAMPHOROXALIC
DERIVATIVES.

By J. BISHOP TINGLE, Ph.D., F.C.S.,
and
WILLIAM EDWIN HOFFMAN, Jun., Ph.D.

(Continued from p. 113).

AMINE DERIVATIVES OF CAMPHOROXALIC ACID.

I. *β*-Naphthylamine.

1. *β*-Naphthylamine (6.4 grms. = 2 molecules) and camphoroxalic acid (5 grms. = 1 molecule) were mixed in boiling alcoholic solution, which was then kept at about 100° on a water-bath for a few minutes. Fine yellow needles soon began to separate out, and, on cooling, a mass of such crystals was deposited. The yield was almost quantitative. The compound is fairly readily soluble in hot alcohol, and is purified by re-crystallisation from it. The purified compound was deposited in the form of fine needles of a pale yellow colour and melted at 169° with the evolution of a gas. Analysis showed it to be *β*-naphthylamine *β*-naphthylcamphoformeneaminecarboxylate,—

0.1723 grm. substance gave 8.6 cc. N at 18° and 770 m.m.

	N.
Calculated for $C_8H_{14} \begin{array}{c} \diagup C : CCOONH_3C_{10}H_7 \\ \diagdown CO \quad \\ \quad NHC_{10}H_7 \end{array}$	.. 5.66
Found	.. 5.74

This salt when treated in alcoholic solution with ferric chloride did not give the violet-red colour characteristic of the enol grouping.

2. When the above compound is heated with a solution of sodium hydroxide, it dissolves, forming sodium *β*-naphthylcamphoformeneaminecarboxylate, and liberating *β*-naphthylamine, which is deposited on cooling the liquid, and was identified by its melting-point, 112°. On acidification of the solution of the sodium salt with dilute hydrochloric acid, the free *β*-naphthylcamphoformeneaminecarboxylic acid is precipitated. It was purified by re-crystallisation from benzene, and is deposited in yellow needles, melting at 172.5–173°. This corresponds to the melting-point obtained for the same compound which was prepared by J. Bishop Tingle (*Journ. Am. Chem. Soc.*, 1901, xxiii., 375). The acid dissolves readily in sodium carbonate solution with the evolution of carbonic anhydride, and in alcoholic solution produces no red colour with ferric chloride.

3. When *β*-naphthylamine *β*-naphthylcamphoformeneaminecarboxylate is heated above its melting-point, it gives off a gas which was proved to be carbonic anhydride by the production of barium carbonate when it was led into a solution of barium hydroxide. A white solid sublimes into the upper part of the tube in which the fusion is being made, and by its melting-point was shown to be *β*-naphthylamine. The fused material, after the evolution of gas had ceased, is purified by means of alcohol, and crystallises in pale yellow needles melting at 173°. Analysis showed it to be *β*-naphthylcamphoformeneamine.

0.1783 grm. substance gave 7.1 c.c. N. at 12° and 760 m.m.

	N.
Calculated for $C_8H_{14} \begin{array}{c} \diagup C : CH \\ \diagdown CO \quad \\ \quad NHC_{10}H_7 \end{array}$	.. 4.59
Found	.. 4.73

With ferric chloride in alcoholic solution no violet-red colour was produced. When *β*-naphthylcamphoformene-

aminecarboxylic acid is heated above its melting-point it gives off carbonic anhydride, and forms a compound melting at 173°, identical with that formed from the *β*-naphthylamine *β*-naphthylcamphoformeneaminecarboxylate by heating.

II. *Paratoluidine*.

1. Camphoroxalic acid (5 grms. = 1 molecule) and paratoluidine (4.8 grms. = 2 molecules) were mixed in boiling alcoholic solution, which, on cooling, deposited a mass of fine yellow needles. The yield seemed to be nearly quantitative. The compound is soluble in alcohol, and is purified by re-crystallisation from it. The purified compound crystallises in slender yellow needles, and melts at 152° with the evolution of a gas. With ferric chloride in alcoholic solution it gave no colour reaction. Analysis proved it to be *paratoluidine paratolylcamphoformeneaminecarboxylate*.

0.1818 grm. substance gave 10.2 c.c. N at 13° and 765 m.m.

	N.
Calculated for $C_8H_{14} \begin{array}{c} \diagup C : CCOONH_3C_6H_4CH_3 \\ \diagdown CO \quad \\ \quad NHC_6H_4CH_3 \end{array}$	.. 6.66
Found	.. 6.67

2. When the above compound is heated with sodium hydroxide solution it dissolves, forming sodium *paratolylcamphoformeneaminecarboxylate*, which, on acidification with dilute hydrochloric acid, precipitates the free *paratolylcamphoformeneaminecarboxylic acid*; this is insoluble in water, and forms a pale yellow mass. The substance, after re-crystallisation from benzene, is deposited in yellow needles, and melts at 168° with the evolution of a gas. It dissolves in sodium carbonate solution with the liberation of carbonic anhydride, and with an alcoholic solution of ferric chloride produces no colour. Analysis showed it to be *paratolylcamphoformeneaminecarboxylic acid*.

0.1605 grm. substance gave 6.2 c.c. N at 6° and 770 m.m.

	N.
Calculated for $C_8H_{14} \begin{array}{c} \diagup C : CCOOH \\ \diagdown CO \quad \\ \quad NHC_6H_4CH_3 \end{array}$	.. 4.47
Found	.. 4.64

3. When the *paratoluidine paratolylcamphoformeneaminecarboxylate* is heated above its melting-point it gives off a gas which was proved to be carbonic anhydride by the production of barium carbonate, when it was led into a solution of barium hydroxide. When the tube in which the fusion is being made is allowed to cool, a white crystalline solid appears in its upper part; this by its melting-point, 42.5°, was demonstrated to be *paratoluidine*. The fused material, after the evolution of gas had ceased, was re-crystallised from alcohol, and is deposited in very pale yellow crystals melting at 178°. With alcoholic ferric chloride no colour is produced. Analysis showed it to be *paratolylcamphoformeneamine*.

0.1710 grm. substance gave 7.8 c.c. N at 12° and 755 m.m.

	N.
Calculated for $C_8H_{14} \begin{array}{c} \diagup C : CH \\ \diagdown CO \quad \\ \quad NHC_6H_4CH_3 \end{array}$	.. 5.20
Found	.. 5.37

When *paratolylcamphoformeneaminecarboxylic acid* is heated above its melting-point it gives off carbonic anhydride, and forms a compound melting at 178°, identical with the one just described, which is formed by heating the *paratoluidine* salt of this acid.

(To be continued).

* From the *American Chemical Journal*, xxiv., No. 3.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, March 1st, 1906.

Mr. A. G. VERNON HARCOURT, F.R.S., Past President, in the Chair.

MESSRS. A. T. Braid, H. E. Fierz, W. P. Hayworth, H. A. D. Neville, F. W. Walker, E. Wechsler, and J. L. White were formally admitted Fellows of the Society.

Certificates were read for the first time in favour of Messrs. Edwin Rodney Chrystall, Forest Villa, Prince's Road, Buckhurst Hill, Essex; Robert K. Duncan, University of Kansas, U.S.A.; Ernest Feilmann, B.Sc., Ph.D., F.I.C., 2A, Dartmouth Road, Brondesbury, N.W.; James Louis Foucar, B.Sc., 20, St. John's Park, Blackheath, S.E.; Richard John Hall, M.Sc., F.I.C., "Sandy-croft," Serpentine Road, Egremont, Liverpool; Walter Daly Scouller, B.Sc., 7, Stamp Office Place, Wakefield; James Frederick Spencer, M.Sc., Ph.D., 134, Rice Lane, Walton, Liverpool; Francis W. Storey, B.Sc., 42, Ellerdale Street, Lewisham, S.E.; George Augustus Turner, Alma House, Strandtown, Belfast.

A certificate has been authorised by the Council for presentation to ballot under By-law I. (3) in favour of Hem Chandra Dutt Gupta, "Guptanibar," Krishnagar, Bengal, India.

Of the following papers, those marked * were read:—

* 45. "*Studies of Dynamic Isomerism. Part IV. Stereoisomeric Halogen Derivatives of Camphor.*" By THOMAS MARTIN LOWRY.

Measurements were given of the solubility in alcohol of α -chloro- and α -bromo-camphors, α 3- and α α -dibromo-camphors, and α 8- and α α -chlorobromocamphors, both alone and in presence of a small proportion of sodium ethoxide. In each case it was found that the addition of alkali resulted in an increase of solubility in the ratio of about 0.9 to 1. No such increase was observed in the case of β -bromocamphor, or of compounds in which both the α - and α' -hydrogen atoms were displaced. The increase of solubility is ascribed to the formation in the solution of a small proportion (7 to 12 per cent) of the stereoisomeric α' -compound (compare Kipping, *Proc.*, 1905, xxi., 124 and 125).

* 46. "*The Coagulating Action of Colloids.*" (Part I.) By WILLIAM PORTER DREAPER and ALEXANDER WILSON.

The addition of tannic acid to an organic colloid like gelatin or albumin in the hydrosol state determines the amount of gallic acid absorbed by the coagulum. The gallic acid may be present before or after the addition of tannic acid. Acids like hydrochloric or acetic reduce this absorption. Salts, on the other hand, increase it. These acids do not seem to replace the gallic acid by mass action or otherwise.

Gelatin in the hydrogel state absorbs gallic acid, although no precipitation takes place if the former is in the hydrosol state. Salts increase absorption, but alcohol reduces it.

Albumin absorbs gallic acid when precipitated by either tannic acid or heat. Alcohol prevents this action and also the absorption of tannic acid by albumin. Addition of gallic acid to a "solution" containing tannic acid and albumin causes rapid coagulation. This reaction is dependent on concentration. In very concentrated solutions, gallic acid will precipitate albumin.

Absorption by silk is greatly reduced in presence of alcohol, the tannic acid absorbed is reduced from 15 to 1.2 per cent. Hide powder behaves in a similar way, the tannic acid absorbed being reduced from 72 to 10 per cent.

Solutions of gelatin, "collin," and albumin give these reactions.

The results obtained by the author throw further light on dyeing and tanning processes. The influence of gallic acid

in the manufacture of leather seems to be of a more direct nature than was previously supposed.

DISCUSSION.

Mr. DREAPER, in reply to questions from Dr. Cain, stated that "collin" was a form of soluble gelatin used by Parker and Payne in the estimation of tannic acid; it did not enter the hydrogel state at the ordinary temperature.

The tannic and gallic acids were estimated by the process he had suggested some time ago. The tannic acid was titrated with copper sulphate solution in the presence of calcium carbonate. The separation of the two acids was brought about by precipitating the former as lead tannate in the presence of a definite excess of acetic acid, which prevented the gallate being precipitated at the same time. The gallic acid in the filtrate was then estimated as copper salt.

* 47. "*Studies on Optically Active Carbimides. III. The Resolution of α -Phenyl- α' -4-hydroxyphenylethane by means of *l*-menthylcarbimide.*" By ROBERT HOWSON PICKARD and WILLIAM OSWALD LITTLEBURY.

A method of resolving racemic hydroxyl compounds by means of *l*-menthylcarbimide was described. The *l*-menthylcarbimides thus obtained by the combination of these compounds can be separated by fractional crystallisation and are then hydrolysed by alcoholic sodium hydroxide. *d*- α -Phenyl- α' -4-hydroxyphenylethane *l*-menthylcarbimate separates in a pure condition from light petroleum and melts at 117°. When hydrolysed, it yields *d*- α -phenyl- α' -4-hydroxyphenylethane, which melts at 64° and has $[\alpha]_D + 7.78^\circ$ in benzene.

DISCUSSION.

Dr. A. MCKENZIE asked whether the authors had obtained any evidence of the partial resolution of an inactive phenol into its optical isomerides by the action of an amount of *l*-menthylcarbimide insufficient for complete interaction. A partial resolution might be expected to occur under such conditions if the velocity of interaction of the *d*-phenol with the *l*-carbimide were different from that of the *l*-phenol. That an inactive alcohol may be partially resolved into its optical isomerides has been shown with *sec*-octyl alcohol (*Ber.*, 1901, xxxiv., 474).

It would be interesting to know whether the temperature conditions under which the fractional crystallisation described by the authors took place had much influence on the progress of the resolution.

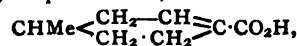
Dr. PICKARD, in reply to a question from Dr. Forster, stated that the method of obtaining carbimides by the action of nitrous acid on the corresponding carbamides could not be used for the preparation of *l*-menthylcarbimide, as this reacts too readily with water.

In reply to Dr. McKenzie, he said that in attempting the resolution of α -phenyl- α' -4-hydroxyphenylethane by treatment with half its equivalent of *l*-menthylcarbimide, some experimental difficulties supervened and led to the adoption of the easier method of fractionally crystallising a mixture of the two modifications of the *l*-menthylcarbimides. No evidence of a transition temperature was observed during the investigation.

* 48. "*Experiments on the Synthesis of the Terpenes. Part VIII. Synthesis of the Optically Active Modifications of Δ^1 -*p*-menthenol(8) and Δ^3 : Δ^8 (9)-*p*-menthadiene.*" By FRANCIS WILLIAM KAY and WILLIAM HENRY PERKIN, jun.

The terpenes and their derivatives, which have so far been obtained synthetically (*Trans.*, 1904, lxxxv., 654; 1905, lxxxvii., 639, 655, 661, 1067, and 1083; *Proc.*, 1905, xxi., 255), have always been inactive, but the following optically active members belonging to this group have now been synthesised.

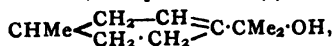
Δ^1 -Tetrahydro-*p*-toluic acid,—



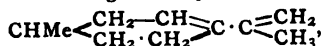
contains an asymmetric carbon atom, and by the systematic

crystallisation of its brucine and strychnine salts the authors have resolved it into its dextro- and levo-modifications. The levo-modification obtained from the brucine salt has a rotation of $[\alpha]_D -100^\circ$, whereas the dextro-acid obtained from the strychnine salt has a corresponding rotation of $[\alpha]_D +101^\circ$.

The esters of these acids were prepared by the action of alcoholic sulphuric acid; ethyl d- Δ^1 -tetrahydro-p-toluate boiled at $154^\circ/100$ m.m. and had $[\alpha]_D +86^\circ$, the levo-modification boiled at $154^\circ/100$ m.m. and had $[\alpha]_D -84^\circ$. On treating the latter with the theoretical amount of magnesium methyl iodide and then decomposing with water and dilute acid, l- Δ^1 -p-menthenol (8),—



was obtained; this substance boils at $101^\circ/14$ m.m. and has $[\alpha]_D -67^\circ$. When this menthenol is digested with potassium hydrogen sulphate, partial racemisation takes place, and the resulting $\Delta^3:8(9)$ -p-menthadiene,—



which distils at $185^\circ/748$ m.m., has a rotation $[\alpha]_D$ of only -10° .

Pure d- $\Delta^3:8(9)$ -p-menthadiene was, however, obtained by the direct action of excess of magnesium methyl iodide on the d-ester without the aid of the acid sulphate; it boils at $184-186^\circ/756$ m.m. and has $[\alpha]_D +98^\circ$. The magnetic rotations and other physical constants of these interesting substances are being determined.

*49. "Studies in the Acridine Series. III. The Methylation of Chrysaniine." By ALBERT ERNEST DUNSTAN and JOHN THEODORE HEWITT.

The authors have continued the research on the lines laid down by Hewitt and Fox (*Trans.*, 1904, lxxxv., 529, and 1905, lxxxvii., 1058), and have prepared the diacetyl and tetra-acetyl derivatives of chrysaniine, starting from the purified base.

By treatment with methyl iodide and methyl alcohol, the diacetyl derivative afforded an acridinium methiodide, which on hydrolysis immediately gave an anhydro-base; this substance was also prepared by the use of methyl sulphate and subsequent hydrolysis and precipitation with ammonia.

50. "Note on the Application of the Electrolytic Method to the Estimation of Arsenic in Wall Papers, Fabrics, &c." By THOMAS EDWARD THORPE.

Known quantities of the material, as a rule about 2 grms., are moistened with lime water, mixed with calcined magnesias, dried, and charred. The ash is treated with dilute sulphuric acid, half a grm. of potassium metabisulphite added, the solution boiled and diluted to a definite volume, and the whole or an aliquot portion, depending on the amount of arsenic suspected to be present, is added to the electrolytic arrangement already described (*Trans.*, 1903, lxxxiii., 974). The deposit of arsenic so obtained is then compared with deposits obtained in precisely the same way by the addition of known quantities of arsenic, ranging from 0.005 to 0.0125 milligram of arsenious oxide, to materials free from arsenic, as in the method of testing brewing materials for arsenic already described (*loc. cit.*). A single incineration of the suspected material suffices for several tests. The amounts of lime-water and magnesia recommended to be used have been proved by direct experiment to retain amounts of arsenic ranging from 0.0025 to 5 milligrams when contained in 2 grms. of wool or paper.

Many samples of woollen materials met with in commerce contain very notable quantities of arsenic, arising probably from the widespread use of arsenical dips.

51. "Nitrogen Halides from Camphoryl- ψ -carbamide." By MARTIN ONSLOW FORSTER and HANS GROSSMAN.

The action of potassium hypobromite and hypochlorite on camphoryl- ψ -carbamide has been found to give rise to dihalogen derivatives which have all the properties of com-

pounds containing halogen attached to nitrogen. Normal camphorylcarbamide yields the same substances, owing to the readiness with which it changes into the *pseudo*-modification under the influence of alkalis. Corresponding monohalogen derivatives have been obtained from camphoryl-methyl- ψ -carbamide and its normal modification.

52. "The Relation of Position Isomerism to Optical Activity. VI. The Rotation of the Menthyl Esters of the Isomeric Chloronitrobenzoic Acids." By JULIUS BEHREND COHEN and HENRY PERCY ARMES.

It was pointed out in a previous paper on this subject (*Trans.*, 1905, lxxxvii., 1192), that whereas chlorine and bromine atoms in the ortho-position to the active group lower its rotation, the nitro-group has the opposite effect of greatly increasing it. In the present investigation, the combined effect of halogen and nitro-group on the activity of the menthyl group has been examined. The authors have studied the rotations of eight out of the ten isomeric chloronitrobenzoic esters, the 2-chloro-3-nitro- and 3-chloro-2-nitro compounds being omitted owing to difficulties of preparation.

Annual General Meeting.

The Annual General Meeting of the Society for the election of Officers and Council and other business will be held on Friday, March 30th, 1906, at 5 o'clock in the afternoon.

The PRESIDENT will deliver his address entitled "The Living Organism as a Chemical Agency: A review of some of the Problems of Photosynthesis by Growing Plants."

SOCIETY OF CHEMICAL INDUSTRY (LONDON SECTION).

Ordinary Meeting, March 5th, 1906.

Mr. A. G. SALAMON in the Chair.

"The Ignition of Nitro-compound Explosives in Small Arm Cartridges." By W. D. BORLAND.

The action of the igniter, i.e., the percussion cap, is to eject through the fire-holes of the case a mixture of solid and gaseous products at temperatures between 2400° C. and 3200° C. in such quantity, volume, and time that the initial resistance of the bullet or shot is overcome before the bulk of the charge of powder develops its full energy, but without any hesitation which may upset alignment or perceptible "hang fire." The rapidity with which these gaseous and solid matters are applied to the powder is determined by exploding the percussion cap in a hollow steel cylinder provided with a hardened steel plunger, and resting upon a crusher lead. The proportion which the crushing pressure bears to total energy is found in practice to be a trustworthy guide to the rapidity with which the heat of the igniter is applied to the explosive, and consequently to the ratio which chamber pressures in the small arm bear to observed velocity of projectile.

The volume of the gaseous matters in relation to the surface of the explosive can be readily determined. These must be large enough to ensure sufficiently high chamber pressures being set up for the most efficient combustion of the powder.

The temperature of ignition was determined by radiation methods of observation, the cap being exploded into a glass tube and the radiation intensity of the solids observed by comparison with a radiant of known intensity, the portion of the spectrum chosen being in the neighbourhood of 6563 wave-length.

The paper includes tables illustrating the action of different igniters on different explosives, both sporting and military, and tracing the effect of total heat energy and temperature of igniter upon velocity, pressure, and rapidity of ignition observed in ballistic trials.

NOTICES OF BOOKS.

Experimental Electrochemistry. By N. MONROE HOPKINS, Ph.D. London: Archibald Constable and Co., Ltd. 1905.

THIS book displays considerable originality in many respects, and teachers of electrochemistry will undoubtedly be able to gather useful hints from it. It is intended to be used both as a theoretical text-book and as a practical laboratory guide, and it is for the latter purpose that it will probably be most appreciated. The descriptions of the various experiments are clear and explicit, and should serve to inculcate a careful spirit in the worker, who is duly warned of the importance of paying attention to those minutiae of practice which are sometimes overlooked. Although the practical notes and hints are particularly valuable, the more theoretical parts of the subject are very satisfactorily treated, the discussion and defence of the theory of electrolytic dissociation being specially worthy of remark, while another valuable feature is the provision of suggestions for additional experiments and for research work, which are introduced at various stages throughout the book. The range of subjects treated is wide, and they are selected in such a way as to make the reader familiar with almost all the important and fundamental work that has been done in most branches of electrochemistry; chapters on the electric furnace, on the preparation of organic compounds, and on the production of nitric acid from the atmosphere being included. The diagrams are clear, and no difficulty should be experienced in setting up the apparatus with their assistance.

Les Erreurs de la Science. ("The Errors of Science"). By LOUIS CHARLES EMILE VIAL. Paris: The Author. 1905.

It has been left to the author of this work to discover that the hypotheses of science are all false, and that its facts are wrongly interpreted, and to propound a new theory of the universe. The author's earlier works, of which this book is an amplification, have met with the reception which, judging from this specimen, they fully deserve, and the author has yet to learn that sincere convictions and a profound belief in his own achievements are not the only equipment necessary for the successful writer of a criticism of the whole region of natural and moral science. It would be profitless to discuss the theories put forward and the supposed results obtained, and it is difficult to believe that even those whom curiosity impels to open the book will be able to read more than a few pages before throwing it aside, thoroughly exasperated by the errors of judgment, the exaggerations and false reasoning displayed in it.

Über die Oxydation des Stickstoffs in der Hochspannungsflamme. ("The Oxidation of Nitrogen in the High Potential Flame"). By Dr. Phil. JOHANNES BRODE. Halle-a-S.: Wilhelm Knapp. 1905.

THIS monograph on the oxidation of nitrogen at a very high temperature discusses the theory of the process, and summarises the work which has been done on the subject from the time of the first experiments of Bunsen and Kolbe, performed when they were investigating the products of the explosion of detonating gas and the consequent fixing of nitrogen, to the present day. The researches of Muthmann, Hofer, and Nernst are very fully described, and publications dealing with the oxidation of nitrogen, both by means of chemical reactions and by the electric discharge, are carefully abstracted. The second half of the monograph is devoted to the discussion of the author's own experiments carried out during the years 1904-1905 at the Institute of Physical Chemistry and Electrochemistry attached to the Technical High School at Karlsruhe. The most suitable means for producing the flame, and the

dependence of the concentration of the nitric oxide formed upon various factors are described in this part of the book, the results obtained by making alterations in the material of the electrodes, their distance apart, the strength of the current, moisture, temperature, &c., being considered at some length.

L'Année Electrique. ("The Electrical Year"). By Dr. FOVEAU DE COURMELLES. Paris: Ch. Béranger. 1906.

THIS annual review of electrical progress is chiefly occupied with the practical applications of the science, special attention being paid to the employment of electrical methods in medicine, though other branches are not altogether neglected. Electrotherapeutics and radiography fill a good portion of the volume, which gives a very readable review of advances made in the year 1905. A great defect in the book is the total absence of all references to original papers, a list of which would double its value. The various articles are, generally speaking, abstracted without comment, and no information is given as to where the original was published for the benefit of those who want further details.

CORRESPONDENCE.

THE ANALYSIS OF DOLOMITE.

To the Editor of the Chemical News.

SIR,—In the CHEMICAL NEWS of December 1, 1905 (vol. xcii., p. 259), Messrs. Clowes and Coleman express their disagreement with our methods described in a previous communication as to the estimation of certain constituents of dolomite (CHEMICAL NEWS, vol. xcii., p. 108). We would say that we have further experimented with the estimation of silica in dolomites, limestones, siderites, and some other minerals, and we have found that, as a rule, either no silica or an inappreciable amount goes into solution after dissolving in hydrochloric acid. At any rate, an evaporation of the hydrochloric acid filtrate does not yield silica, and if there is any in solution it would be necessary to seek it in a later stage of the analysis in connection with iron and alumina. The results of our experiments will be published at some future time.

With regard to the value of (A), a double precipitation of iron and alumina, as against (B), the single precipitation, we obtain the following results:—

	(A). Double precipitation.	(B). Single precipitation.
	Per cent.	Per cent.
1.	0.56	0.59
2.	—	0.545

and with a second specimen—

	(A). Double precipitation.	(B). Single precipitation.
	Per cent.	Per cent.
1.	0.47	0.49
2.	—	0.47

We still maintain that if one is careful to use only a small excess of ammonium hydrate in the precipitation, even though the ammonia contains some ammonium carbonate, a double precipitation is not necessary. It is unnecessary to add more than a drop or two in excess, as the iron present serves as a perfect indicator. The very small amount of calcium carbonate thus precipitated would doubtless be removed by properly washing the precipitate.

Messrs. Clowes and Coleman obtained in a certain specimen of dolomite 1.43 per cent of iron and alumina by the single precipitation, and 1.35 per cent by the double precipitation. They infer that 0.08 per cent of calcium is precipitated in the first instance. That seems like a rather small difference to obtain by two distinct methods, especially in view of the fact that it is not easy to obtain exactly concordant results by the same method.

A competent authority (Washington, "Rock Analysis") says:—"No method is capable of yielding results of absolute accuracy." If one were to make determinations of iron in the same specimen, for example, by permanganate or bichromate titration, would he not be satisfied with results that varied from one another by within 0.1 per cent?

A method of estimating carbon dioxide in a mineral or rock is described in a well-known and valuable work (Crookes, "Select Methods," p. 600), where it is claimed that by using 5 grms. of Iceland spar the method will give 43.97 per cent of carbon dioxide, 44 per cent being the theoretical.

The degree of concordance of course depends on the nature of the analysis. Results of the analyses of organic substances by combustion are often published when they differ by 0.30 per cent to 0.50 per cent from one another, or from the theoretical. An authority suggests (Washington, "Rock Analysis"), if in the analysis of a rock the sum of the constituents found aggregate from 99.50 per cent to 100.75 per cent, the results can be accepted as satisfactory.

Can it be certain that a sample of rock is usually mixed so thoroughly that two portions from the same would be exactly identical? If this could be assumed, the set of weights used would have to be very finely calibrated to give identical results. Then there looms up the possibility that the distilled water may not be perfect, and the reagents employed may not be absolutely pure, and even if they are all that could be desired, they might attack the glass, all of which works against perfect results. If, therefore, Messrs. Clowes and Coleman obtain results by two different methods that vary by only 0.08 per cent, would it not be an argument in favour of the simpler and easier method?

Concerning the importance of removing the ammonium salts before precipitating the magnesium, Hillebrand (*Bull. U.S. Geol. Survey*, p. 64) says:—"It is not necessary to first remove ammoniacal salts unless very little magnesium is present, and then only to hasten precipitation." Newbauer (*Zeit. für Angew. Chem.*, 1896, p. 435) has shown that precipitation is complete, even in presence of large quantities of salts of ammonium, including the oxalate. Messrs. Clowes and Coleman also generously admit that "the difference in the percentage of MgO is not so important."

The writer acknowledges his indebtedness to Mr. A. A. Wells for assistance in the experimental work before described.—I am, &c.,

NICHOLAS KNIGHT.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances de l'Académie des Sciences. Vol. cxlii., No. 7, February 12, 1906.

Re-combination of Ions of Saline Vapours.—G. Moreau.—On account of the value of their mobility, as well as their coefficient α , the ions of saline vapours between the temperatures of 170° and 0° lie between the ions of ordinary gases and the total ions due to the oxidation of phosphorus. In proportion as the temperature rises their mass diminishes, and when heated in a flame these ions become comparable with particular cathodic particles for the negative ion, and the atom of hydrogen for the positive ion.

Calcium Iodomercurates.—A. Duboin.—A saturated solution of mercury iodide in calcium iodide when filtered is of density 2.89, and has a composition represented by the formula $\text{CaI}_{2.130}\text{HgI}_{2.123}\text{H}_2\text{O}$. When left to cool, a deposit of very voluminous yellow crystals comes down,

which are extremely deliquescent, and correspond to the formula $\text{CaI}_2.\text{HgI}_2.8\text{H}_2\text{O}$, and have density 3.258 to 3.337. A second iodomercurate may be obtained from the same solution in the form of very small crystals, and having formula $\text{CaI}_{2.5}\text{HgI}_2.8\text{H}_2\text{O}$. A third one, by cooling the solution below 0°, appears in the form of long prisms having formula $3\text{CaI}_{2.4}\text{HgI}_2.24\text{H}_2\text{O}$ and density 3.56 to 3.66.

Phosphorus Sulphides.—H. Giran.—The existence of a certain number of phosphorus sulphides is still under discussion. An investigation of the temperature of fusion of mixtures of varying proportions of sulphur and phosphorus enables the author to make some definite statements on this subject. His researches are all made on mixtures which were previously heated to a higher temperature, and in which there was certainly a union of the two non-metals. He finds that mixtures of sulphur and phosphorus, which are liquid at ordinary temperatures, show very markedly the phenomenon of surfusion.

Preparation and Properties of Strontium.—MM. Guntz and Roederer.—The authors prepare strontium in the same manner as M. Guntz previously prepared barium. First, strontium hydride is prepared quite free from mercury by the action of hydrogen on strontium amalgam. In the vacuum of a mercury pump, at about 1000°, this compound dissociates, and the strontium vapour can then easily be condensed on a cold steel tube. The properties of this substance, which contains 99.43 per cent of pure strontium, are then examined.

Action of certain Bibasic Acid Ethers on the Halogen-magnesium Derivatives of the Primary Aromatic Amines.—F. Bodroux.—The author previously showed that neutral ethyl carbonate reacts on the halogen-magnesium derivatives of the primary aromatic amines, giving rise to the mono-substituted derivatives of urethane. If neutral ethyl oxalate is used, a symmetric disubstituted oxamide is produced, and with excess of the ethyl oxalate a very small quantity of substituted oxamic ether is formed, $\text{CO}-\text{NH}-\text{R}$.

The author obtains, with good yields, COOC_2H_5 , diphenyloxamide, ortho- and para-dicresyloxamides, and, with a bad yield, β -dinaphthyloxamide.

Constitution of Chromic Sulphates.—Albert Colson.—The green sulphate resulting from the reduction of a cold solution of chromic acid by means of sulphur dioxide has for its composition after desiccation in a vacuum $\text{Cr}_2(\text{SO}_4)_3 + 7.5\text{H}_2\text{O}$. It contains two concealed SO_4 radicals, and is transformed into a turquoise-blue salt, $\text{Cr}_2(\text{SO}_4)_3 + 10\text{H}_2\text{O}$, which contains only one concealed radical. These two bodies are intermediate between the ordinary violet sulphate, of which the entire acid acts on barium chloride, and a unknown sulphate of which the acid is entirely concealed, and of which the author in this present research establishes the existence.

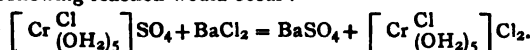
Existence of Bicarbonates in Mineral Waters, and supposed Anomalies in their Osmotic Pressures.—L. C. Maillard and Lucien Graux.—The authors calculate individually the part which each of the constituents of a mineral water plays in the establishment of the osmotic pressure, and they find that, in the cases examined, the cryoscopic results offer no opposition to the admitted notion of the existence of bicarbonates in the waters. Further, a judicious application of the laws of cryoscopy to mineral waters dispels the supposed anomalies of the osmotic pressure.

Berichte der Deutschen Chemischen Gesellschaft.
Vol. xxxviii., No. 2, 1906.

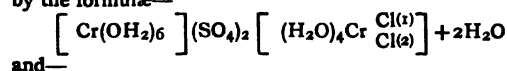
Autoxidation of Trivalent Titanium.—W. Manchot and P. Richter.—If caustic potash is added to a salt of trivalent titanium and the precipitate of titanium sesquioxide obtained is shaken up with oxygen, oxidation speedily

takes place, a considerably greater volume being absorbed than corresponds to one equivalent of oxygen. On being acidified the solution becomes yellow. Hydrogen peroxide first results, but it is again partially reduced by the titanous oxide, and forms pertitanic acid only on acidifying, or possibly even in the alkaline liquid. To explain this process by a quantitative experiment it was first of all necessary to prevent the secondary action of the hydrogen peroxide in the alkaline liquid. This was done by using a fairly thick paste of lime instead of caustic potash. A solution of titanium sesquioxide was prepared by reducing a sulphuric acid titanium dioxide solution with zinc. A hydrochloric acid solution, produced by the action of barium chloride upon the sulphuric acid solution could also be employed. These solutions rapidly undergo a change in air, but may be kept for some time in a flask filled with hydrogen. Due precautions were taken to prevent the oxidation of the titanium before mixing with alkali, and the end of the oxidation was recognised by the complete decoloration of the precipitate. It was found that all the oxygen taken up was quantitatively converted into hydrogen peroxide. The latter is totally precipitated by the lime. Not only is the action of the hydrogen peroxide on the unoxidised titanium hindered by the lime, but also the formation of pertitanic acid cannot result under these conditions until after acidification; *i.e.*, secondarily. The direct formation of TiO_3 would require the absorption of three equivalents of oxygen.

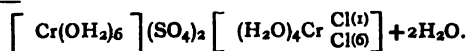
Chromium Salts.—A. Werner and R. Huber.—Recoura has described a chlorosulphate of chromium to which he gave the formula $[Cr^{Cl}(OH_2)_5]SO_4 + xH_2O$. The authors hoped that when this was treated with barium chloride the following reaction would occur:—



They found, however, that under these circumstances a green solution was obtained, which after concentration gave the original chlorosulphate on addition of sulphuric acid, but when they tried to obtain the supposed new chloride, $[Cr^{Cl}(OH_2)_5]Cl_2$, in the pure state they found that it was not a single compound, but a mixture of green and blue chromium chlorhydrate. Hence they supposed that Recoura's chlorosulphate contained a hexaquo-chrom complex, and this assumption was confirmed as follows:—If the chlorosulphate was exposed to moist air upon a clay plate for some time, its blue-green colour altered, and finally a reddish violet powder consisting of violet chromium sulphate remained behind. Hence it must be concluded that Recoura's chlorosulphate has not the simple formula, but it is probably a di-molecular compound $Cr(OH_2)_6 \begin{smallmatrix} SO_4 \\ SO_4 \end{smallmatrix} [Cr^{Cl_2}(OH_2)_2]_2$. This assumption may be proved synthetically by adding concentrated sulphuric acid drop by drop to an ice-cold mixture of concentrated solutions of violet chromium sulphate and green chromium chloride, $[Cr^{Cl_2}(OH_2)_4]Cl + 2H_2O$. A copious precipitate of green crystals is obtained which have the same composition as Recoura's chlorosulphate and behave similarly, but there are certain differences in the crystalline form and colour of the two compounds. Possibly these differences may be explained by assuming a spacial difference in the complexes of the two salts, $[Cr^{Cl_2}(OH_2)_4]$, as represented by the formulæ—



and—



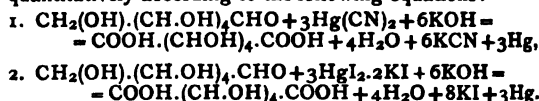
This assumption must be accepted with caution, and it is important to note that neither Recoura's chlorosulphate

nor the synthetic compound gives a precipitate of silver chloride when silver nitrate is added to the freshly prepared neutral aqueous solution.

Aldehydes as Acids.—H. Euler.—It has been shown that formaldehyde is a weak acid, whose dissociation constant amounts to $1 \cdot 10^{-14}$ at 0° , and that with alkalis it forms salts which are half hydrolysed in normal solution. The author has examined other aldehydes—chloral hydrate, *d*-fructose, *d*-glucose, acetaldehyde, and furfural—and finds that they all behave like acids, their dissociation constants being $4 \cdot 10^{-12}$, $3 \cdot 6 \cdot 10^{-13}$, $1 \cdot 8 \cdot 10^{-13}$, $0 \cdot 7 \cdot 10^{-14}$, and less than $1 \cdot 10^{-16}$ respectively. In all probability the formation of salts is a general property of aldehydes, though it cannot always be detected by the means at present at our disposal.

Oxidation of Trivalent Titanium (II).—W. Manchot and P. Richter.—Trivalent titanium behaves like divalent iron when oxidised by molecular oxygen, requiring twice as many equivalents as correspond to the conversion of Ti_2O_3 into $2TiO_2$, but in the case of iron no hydrogen peroxide is formed. Iron exhibits the same peculiarity towards other oxidisers, *e.g.*, chromic acid and permanganic acid. If a solution of chromic acid and hydriodic acid is prepared and a titanium sesquioxide solution added, iodine is liberated, and may be determined by titration with thiosulphate. The experiments show that two equivalents of iodine are liberated, so that altogether three equivalents of oxygen are replaced by one atom of titanium. With permanganate the same result is obtained. If reduced titanium solution is added to a mixture of permanganate and hydrochloric acid, of such strength that it changes only very slowly, a strong smell of chlorine is noticed.

Two New Methods for the Quantitative Determination of Grape-sugar.—B. Glassmann.—1. *Indirect Volumetric Method.*—When glucose is oxidised by means of an alkaline mercury cyanide solution, or a mercury iodide-potassium iodide solution, the oxidation proceeds quantitatively according to the following equations:—



Measured quantities of a grape-sugar solution of known strength are introduced into excess of boiling alkaline mercuric cyanide solution, and the mercury is filtered off, dissolved by heating with concentrated nitric acid, and the mercury in the nitrate thus obtained is determined by Rupp and Krauss' method; *i.e.*, the solution is diluted with water, saturated iron alum solution and nitric acid added, and the mixture titrated with rhodammonium in the usual way till a permanent light brown coloration is obtained.

2. *Gas Volumetric Method.* If mercury cyanide or mercury iodide-potassium iodide is warmed with a hydrazine salt the following reaction occurs quantitatively: $2Hg(CN)_2 + 6KOH + N_2H_4.H_2SO_4 = K_2SO_4 + 4KCN + 2Hg + 2N + 6H_2O$.

If the nitrogen is collected, the amount of mercury in the mercury solution can be calculated from it. If a grape-sugar solution is treated with a known excess of titrated mercury cyanide solution by Method 1 and the excess of mercury is determined by treating the alkaline solution with excess of hydrazine sulphate, the difference gives the mercury separated by the oxidation of the grape-sugar, and from this the strength of the grape-sugar solution can be calculated (3 grms. Hg = 1 molecule $C_6H_{12}O_6$).

Action of Atmospheric Oxygen upon Paracautouchou.—Edgar Herbst.—A solution of purified paracautouchou in benzene was heated for one hundred and forty hours, and treated with atmospheric air which had been freed from dust and moisture, the benzene which evaporated being replaced. During the process the originally colourless solution gradually became yellower and yellower. At the end of the operation a small quantity

of a yellowish grey resinous substance had collected on the bottom of the flask and in the tube through which the air was introduced. This substance was apparently not soluble in alcohol, petroleum ether, glacial acetic acid, or carbon disulphide, but there was not enough of it for a further examination. The benzene solution when filtered off gave no precipitate of caoutchouc on addition of alcohol. When the benzene was evaporated, a syrupy transparent reddish brown mass remained behind. This when treated with petroleum ether went into solution for the most part, leaving a small greyish yellow residue. The petroleum ether solution on evaporation left a perfectly clear syrup of light reddish brown colour and a peculiar aromatic smell. Its composition corresponded to the formula $C_{10}H_{16}O$. The resinous substance, insoluble in petroleum ether, was re-precipitated from benzene solution with petroleum ether, and then yielded an amorphous mass with a faint aromatic odour. Analysis showed that its composition was $C_{10}H_{16}O_3$. The liquids from which this compound, richer in oxygen, was precipitated were collected and evaporated. After repeated precipitation from benzene solution by means of a large excess of petroleum ether, a greasy substance was obtained of empirical formula $C_{10}H_{16}O_3$. It formed a hard mass *in vacuo*. The oxidation product, $C_{10}H_{16}O_2$, could not be prepared.

MISCELLANEOUS.

Royal Institution.—On Tuesday next, March 20, at 5 o'clock, Mr. J. E. Marr delivers the first of three lectures at the Royal Institution on "The Influence of Geology on Scenery" (the Tyndall Lectures); and on Thursday, March 28, at the same hour, Prof. Bertram Hopkinson begins a course of three lectures on "Internal Combustion Engines" (with Experimental Illustrations). The Friday Evening Discourse on March 23 will be delivered by Lord Roberts on "Imperial Defence"; on March 30 by Prof. Zeeman, on "Recent Progress in Magneto-optics"; and on April 6 by Mr. W. B. Hardy, on "The Physical Basis of Life."

Thermoelectric Properties of Alloys.—N. N. Tournour.—The author has examined the thermoelectric properties of alloys of tin with bismuth and lead, of lead with bismuth, and of some alloys of copper with zinc. The electromotive force was measured relatively to copper; one of the junctions was kept at 0° , the other at 100° . He found:—1. That the electromotive force of alloys of tin and lead is constant, and does not depend upon the composition of the alloy; it is the same as that of lead. 2. The electromotive force of alloys of bismuth with tin and lead, and of copper with zinc, varies in amount and even in direction according to the proportion of the components. 3. The maximum electromotive force corresponds with alloys which appear to be definite compounds, $SnBi_3$ and $PbBi_3$. 4. Alloys of lead and bismuth, of copper and zinc, and of zinc and lead are examples of phases in which the electromotive force remains constant and independent of the composition; for example, with lead and bismuth at 65 per cent of lead and over, with copper and zinc between 10 per cent and 25 per cent of zinc. 5. The alloys of copper and zinc have at least two maxima and two minima, so that by varying the composition the direction of the current changes at least four times. The numerical values have not yet been confirmed. 6. With time there is produced in the alloys of bismuth with tin or lead a modification which manifests itself by a diminution of the electromotive force.—*Journ. Soc. Phys. Chim. R.*, vol. xxxvi., p. 1119.

The Halogen Salts of Palladium.—A. Gutbier and A. Krell.—The chloropalladites and the chloropalladates

of the alkaline metals are anhydrous, and their formulæ are respectively $PdCl_4M_2$ and $PdCl_6M_2$; the same is the case with the corresponding bromides. The chloro- and bromopalladates are decomposed by boiling water with the disengagement of chlorine or bromine; the same action takes place with concentrated sulphuric acid. They are also reduced by ammonia with the disengagement of nitrogen. *Chloropalladite of Ammonium*.—We evaporate carefully the mixture $PdCl_2 + 2NH_4Cl$; beautiful yellowish green needles are formed, soluble in water, giving a deep brownish red colour. *Chloropalladite of Cesium*, bright brown precipitate, re-crystallises in warm water in the form of needles. *Bromopalladate of Cesium*, a brownish yellow precipitate almost insoluble in cold water, resulting from the action of chlorine on a solution of chloropalladite. *Chloropalladite of Rubidium* resembles the cesium salt, re-crystallises in the form of silky needles. *Chloropalladate of Rubidium*, an orange coloured microcrystalline precipitate resembling the cesium salt. *Bromopalladite of Ammonium* is obtained in the same way as the double chloride; beautiful reddish brown needles, very soluble even in the cold. *Bromopalladate of Ammonium*.—We cause the vapour of bromine to act slowly on a cold solution of the preceding salt; it forms regular black octahedra, very slightly soluble. *Bromopalladite of Cesium*, reddish brown needles, very soluble. *Bromopalladate of Cesium*, precipitate of black octahedra, prepared in the same manner as the salt of ammonium. *Bromopalladite and Bromopalladate of Rubidium* resemble the corresponding cesium salts. *Bromopalladate of Potassium*, beautiful black octahedra, soluble in cold water, giving a brown coloration.—*Berichte*, vol. xxxviii., p. 2385.

Products of the Higher Oxidation of Chromium.—E. H. Riesenfeld, H. E. Wohlers, and W. A. Kutsch.—The authors have prepared afresh the salts obtained by Hoffmann and Hiendlmaier, and give them the formula CrO_8M_3 instead of CrO_6M_2 . The salt of ammonium, $CrO_8(NH_4)_3$, is prepared by cooling to below 0° a solution of 50 c.c. NH_3 at 25 per cent, and 25 c.c. of CrO_3 at 50 per cent; to 75 c.c. we then add carefully 25 c.c. of H_2O_2 at 30 per cent, and prevent the temperature rising above 0° . The liquid becomes brown, and after a few hours deposits crystals, which are drained and washed several times with alcohol at 95 per cent. In a similar manner the salts of potassium and sodium are prepared. If, on the other hand, we operate in an acid solution, we obtain the blue salts of M. Wiede (for example, for the ammonium salt, $CrO_8H_2NH_4$ we take 100 c.c. of water, 5 c.c. of concentrated HCl , 10 grms. of NH_4Cl , 50 c.c. CrO_3 , and 25 c.c. of H_2O_2 at 30 per cent), only the washing with alcohol should be rapid on account of the solubility of the blue salts. The salts of ammonium or potassium, (CrO_8M_3), form small reddish brown irregular octahedra; the sodium salt occurs in orange-coloured plates. These salts do not undergo any change in absolute alcohol or anhydrous ether even when boiling. If these liquids contain water there may be some change with the formation of aldehyde. Cold water dissolves them slightly, but it decomposes them slowly. Neutral or alkaline solutions soon form chromates and oxygen, which solutions, strongly acidulated with sulphuric acid, form, first of all, perchromic acid, then a chromic salt. The well-dried salts keep better in a slightly moist atmosphere (almost indefinitely, in fact); the sodic salt is the one that undergoes spontaneous decomposition most rapidly. Finally, heat alone decomposes the salt of potassium at about 170° into KOH , CrO_4K_2 , and oxygen. The presence of impurities makes the salts more easily decomposable by heat, sometimes even explosive. When moistened with concentrated sulphuric acid the salts deflagrate, throwing off in all directions green morsels of Cr_2O_3 . The same phenomenon takes place by submitting the salt of ammonium to a shock; there is, further, a violent explosion. The salts of potassium and sodium are insensible to shock.—*Berichte*, vol. xxxviii., p. 1885.

MEETINGS FOR THE WEEK.

- MONDAY, 19th.**—Society of Arts, 8. (Cantor Lectures). "Fire, Fire Risks, and Fire Extinction," by Vivian B. Lewes.
- TUESDAY, 20th.**—Royal Institution, 5. (Tyndall Lectures). "The Influence of Geology on Scenery," by John E. Marr, F.R.S., &c.
Society of Arts, 8. "English Royal Heraldry," by Cyril Davenport, F.S.A.
- WEDNESDAY, 21st.**—Microscopical, 8. "Contribution to our Knowledge of the Rotifera of South Africa," by C. F. Rousselet. "Resolving Limits for the Telescope and the Microscope," by E. M. Nelson.
Society of Arts, 8. "Motor Boats," by B. B. Redwood, B.A.
- THURSDAY, 22nd.**—Royal Institution, 5. "Internal Combustion Engines" (with Experimental Illustrations), by Prof. Bertram Hopkinson, M.A., &c.
- FRIDAY, 23rd.**—Royal Institution, 9. "Imperial Defence," by The Rt. Hon. Bart Roberts, V.C., &c.
Physical, 5. "Unilateral Electric Conductivity over Damp Surfaces," by F. T. Trouton. "Construction and Use of Oscillating Valves for Rectifying High-frequency Electric Currents," by J. A. Fleming. "Use of the Cymometer for the Determination of Resonance Curves," by G. B. Dyke.
- SATURDAY, 24th.**—Royal Institution, 3. "The Corpuscular Theory of Matter," by Prof. J. J. Thomson, F.R.S., &c.

WHARF at RAINHAM.—FREEHOLD.—River frontage to Rainham Creek, near the River Thames, and in the midst of the Industrial Factory district. With possession.

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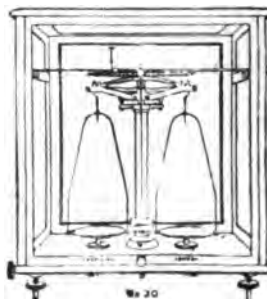
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ROOT-SAP ACIDITY.

By W. F. SUTHERST, Ph.D., F.I.C.

A STANDARD solution to represent the dissolving power of root-sap on mineral matter would be an ideal implement in the hands of agricultural chemists, since, by its help, the amount of plant food in the soil and available matter in mineral manures could be determined with exactitude.

Certain English and foreign chemists have suggested solutions—notably Stutzer, Dehérain, Wagner, Dyer, Hughes, and others—such being based more or less on the acidity of root-sap, and containing either an organic acid or acid salt. It is obvious that there is an acid solution in the roots of all plants, and such would, of course, have a fairly strong solvent action on insoluble phosphates, chalk, potash compounds, &c., found in the soil, and one would expect that the roots containing the larger amounts of acid

contains about the largest percentage of mineral matter (viz., 2.04); while white clover, with an acidity of 1.43 per cent, contains only 1.43 per cent mineral matter. Phosphoric acid, present of course in the soil as insoluble bicalcium or other phosphates, is supposed to be particularly affected by the amount of acid in root-sap; but we see that rye grass, with its low root acidity, takes up slightly more than white clover. One might say that these plants for their growth need only a fixed amount of phosphoric acid, and take up but that quantity; but, during the evolution of these plants, if they in pre-historic times contained such acid sap, they would probably have adapted themselves to the larger quantities of phosphoric acid that they were capable of extracting out of the soil.

Since there is an acid liquid in the roots of all plants, it is probable that plant growing in very chalky soils would be affected by the neutralising action of the chalk, and some experiments were carried out here by me by growing wheat in soils containing various quantities of chalk, the crops being examined when grown for shoots, roots (in green and dry state), root-sap acidity, and mineral matter. The results obtained are given in Table II., the same number of seeds being used in each case.

The first thing noticeable is the diminished yield on the soil rich in chalk, then the amount of roots remaining practically constant; but the increasing proportion of roots

TABLE I.

	Water.	Root acid.	Root-sap acidity.	Ash.	P ₂ O ₅ .	K ₂ O.	Acid : Ash.
Rye grass	70	0.25	0.35	2.04	0.22	0.71	1 : 5.83
Potato (tops) ..	80.3	0.29	0.34	1.97	0.16	0.44	1 : 5.80
Pea	81.5	0.30	0.34	1.39	0.15	0.52	1 : 4.09
Sugar-beet	89.7	0.41	0.48	1.53	0.07	0.40	1 : 3.18
Timothy	70.4	0.50	0.80	2.05	0.24	0.70	1 : 2.56
Turnip (white) ..	89.8	0.46	0.51	1.19	0.08	0.28	1 : 2.33
Spinach	90.3	0.62	0.70	1.60	0.16	0.27	1 : 2.28
Oat	81.0	0.43	0.65	1.42	0.13	0.56	1 : 2.18
Onion	86.0	0.41	0.45	0.80	0.14	0.25	1 : 1.77
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Cabbage	87.1	0.70	1.00	1.40	0.21	0.39	1 : 1.40
Artichoke	81.1	0.56	0.76	1.01	0.39	0.24	1 : 1.35
Red clover	82.0	1.10	1.37	1.55	0.13	0.55	1 : 1.13
White clover ..	80.5	1.00	1.43	1.43	0.18	0.31	1 : 1
Incarnate clover ..	81.5	0.83	1.28	1.13	0.08	0.26	1 : 0.88

TABLE II.

	Chalk in soil.	Shoots.	Roots.	Dry shoots.	Dry roots.	Roots to shoots.	Roots to shoots (dry).	Sap acidity.	Ash in dry matter.
	Per cent.	Grms.	Grms.	Grms.	Grms.				Per cent.
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3.	21.40	109.7	95.0	17.3	15.7	1 : 1.15	1 : 1.13	0.260	7.14

would absorb more mineral matter (the elaborate analyses of Dyer on the acidity of root-sap proved that the amounts of acid in various plants varied considerably), but such is not the case, and is one drawback to using standard solutions based on the acidity of root-sap. On comparing the sap acidity of several plants with the amount of mineral matter they contain, it will be seen that the proportion is the reverse, viz., that the most acid roots bring less mineral matter into the plants. The flow of water through the plant system, due to transpiration, might have some effect in this case, but the amount of water transpired by the plants enumerated in Table I. is about the same, consequently some other reason must be given for this phenomenon. Table I. gives the plant, amount of water in same, amount of acid in the roots (expressed as citric acid), sap acidity—i.e., ratio of root acid to amount of water in roots (these figures taken from Dyer's "Available Mineral Plant Food in Soils"),—ash, phosphoric acid, potash, and proportion of acid to ash. It will be seen from this table that rye grass, with a weak root-sap acidity of 0.35 per cent,

to shoots show that the roots must have been travelling away from the plant base in their search for food, and probably endeavouring to avoid the chalk, for the roots of Nos. 2 and 3 were very much longer than No. 1. The root-sap shows a slight decrease in acidity, due no doubt to the neutralising action of the chalk. In testing the acidity, great care was taken to wash the root free from chalk, and no roots were tested without the washings were quite free from all trace of milkiness. After drying the roots, they were ground to a fine powder and the acid extracted by boiling water and titrated with normal alkali. The small amount of ash in the Nos. 2 and 3 samples also shows the neutralising action of the chalk, as the roots, having absorbed their requisite amount of lime, would most probably cease dissolving out mineral matter, which would be largely lime salts, and the plant cannot show a preference for one mineral substance—such as phosphate—when another is present in overwhelming quantities. Possibly the roots form a crust of calcium oxalate and tartrate when in contact with large quantities of chalk

which prevents the passage of moisture from without and acid sap from within; consequently the roots, in their search for food, try to leave the chalky region.

The Agricultural College, Tanworth.

NOTE ON THE PREPARATION OF BENZONITRILE.

By J. BISHOP TINGLE, Johns Hopkins University.

IN the course of some work which was being carried out in this laboratory in 1904, it was necessary to provide rather large quantities of benzonitrile; at my suggestion, therefore, Messrs. E. J. Hoffmann and W. A. Syme studied some of the various methods which have been proposed for the preparation of this compound. Their results are recorded in the following table; they have been checked in most cases by other students. The figures given for the cost are believed to be accurate *relatively*, the *absolute* cost is necessarily variable. In reckoning the time no account was taken of the washing and distillation of the crude product, as this was practically the same in all cases; the time required to dry an ethereal solution, when this was needful, has also been disregarded, because it did not occupy the worker. The yield refers to material which has been distilled.

Method I.—Sandemeyer's reaction (*Ber.*, xvii., 2653):—Aniline, 35 grms.; copper sulphate crystals, 100 grms.; potassium cyanide, 110 grms.; sodium nitrite, 25 grms.; and hydrochloric acid, 50 grms. were employed.

Method II.—From benzamide, 15 grms.; and phosphoric anhydride, 25 grms. (Hofmann and Buckton, *Ann. Chem. Pharm.*, c., 155).

Method III.—From benzoic acid, 100 grms.; and lead thiocyanate, 240 grms. (Krüss, *Ber.*, xvii., 1766).

Method IV.—From sodium benzenesulphonate, 100 grms.; and potassium cyanide, 100 grms. (Merz, *Ibid.*, iii., 710).

Method V.—From benzamide, 23 grms.; and quicklime in excess (Anschutz and Schulz, *Ann. Chem. Pharm.*, xcvi., 48).

Method VI.—From benzoic acid, 50 grms.; and potassium thiocyanate, 20 grms. (Letts, *Ber.*, v., 673).

As was to be expected Mr. Syme did not obtain a large yield of benzonitrile by the use of iodobenzene and silver cyanide (Merz and Weith, *Ibid.*, x., 746), nor was he able to get satisfactory results by the distillation of aniline oxalate (A. W. Hofmann (*Comptes Rendus*, lxiv., 388). He prepared benzamide very easily and quickly from benzoic acid and ammonium thiocyanate (Kekulé, *Ber.*, vi., 113).

Method.	Yields. Grms.	Per cent of the theoretical.	Time.	Cost of 100 grms.
I.	25	64	About 4 hours	0.72 dol.
II.	12	95	" 1 "	1.00 "
III.	60	71	" 10 "	0.55 "
IV.	15	26	" 1 "	5.86 "
V.	8.2	42	" 1 "	1.70 "
VI.	12.2	58	" 3 "	1.15 "

—*American Chemical Journal*, xxxv., No. 1.

Researches in the Pyrane Series.—E. E. Blaise and H. Gault.—To obtain pyranic homologues, the authors condense oxalacetic ether with various aldehydes, both cyclic and acyclic. The cyclic aldehydes do not give the alcoylidene-bisoxalacetic ethers, but only ketoarylparaconic ethers, on account of the elimination of 1 mol. of alcohol between 1 mol. of aldehyde and a single molecule of oxal-

acetic ether, $\text{CO}-\text{CH}-\text{CO}_2\text{C}_2\text{H}_5$,
 $\text{CO}-\text{O}-\text{CH}-\text{C}_6\text{H}_4-\text{R}$ By effecting the

condensation by means of gaseous hydrochloric acid, keto-paraconic ethers are obtained directly; if, on the contrary, diethylamine is employed, the corresponding salts of diethylamine are formed.—*Comptes Rendus*, cxlii., No. 8.

COMPARISON OF A WET AND CRUCIBLE-FIRE METHODS FOR THE ASSAY OF GOLD TELLURIDE ORES, WITH NOTES ON THE ERRORS OCCURRING IN THE OPERATIONS OF FIRE ASSAY AND PARTING.*

By W. F. HILLEBRAND and E. T. ALLEN.

(Concluded from p. 123).

PART II. THE ERRORS IN GOLD ASSAYING (*continued*).

Retention of Lead by Cupelled Beads,

INASMUCH as in hardly a single case did our corrected assays on proof metal show more than was weighed at the start, even at the lowest heat, with full feather litharge, it did not seem necessary to assume the presence of lead in the beads obtained. Nevertheless, in one case a number of them from the same series of assays were dissolved in aqua regia together, the gold precipitated, and the solution evaporated to a few drops. By exercise of the utmost care it was possible to detect a minute quantity of lead, too small, perhaps, to have influenced the results to any appreciable extent.

Later, the following experiments were carried out:—

Experiment 1.—Three beads of proof gold, weighing, respectively, 30.14, 50.40, and 10.35 m.grms., were cupelled with 2 grms. of lead each, in the ordinary way, and weighed after boiling with dilute hydrochloric acid to remove any film of lead oxide or particles of litharge-soaked bone-ash. They were then dissolved together in aqua regia, evaporated, diluted with sulphurous acid, the solution filtered, and evaporated till fumes appeared. The residue, consisting of lead sulphate and a little metallic gold, was transferred to a filter after taking up with alcohol, the sulphate extracted by ammonium acetate, the lead re-precipitated by hydrogen sulphide, re-converted into sulphate, and weighed. The weight was 0.0004 gm., equivalent to 0.3 per cent of lead.

Experiment 2.—Three beads of very nearly the same weights as those first acted on were treated in a similar manner, except that they were left in a muffle twenty minutes after brightening. The amount of lead recovered from all three together was 0.37 per cent.

This shows clearly, as had our earlier experience, that the error, though of some magnitude, is not lessened by leaving the beads in the muffle for a considerable time after brightening. It should be said that the sulphate weighed was certainly of lead and not silver. The percentages above given are, however, to be regarded as approximate only.

Losses of Gold in Slags.

From a statement in Fulton's paper, "Assay on Telluride Ores" (*Journ. Am. Chem. Soc.*, 1898, xx., p. 589), we were led to expect much greater losses of gold in the slag than appear from the tables in Part I. of this paper. Fulton says that these are much greater with Cripple Creek telluride ores than with ordinary gold ores. They appear, however, so far as our own work goes, to be slight. We have suggested a possible explanation for his higher losses. (See "Discussion of Results," p. 109).

Errors in Parting.

Strength of Acid.

Most diverse is the practice in parting as to the strengths of acid employed. Vauquelin seems to have first used acid of 1.16 and 1.26 specific gravity, and these are the strengths most frequently recommended to-day. But there are not wanting those who prescribe other strengths, or who reverse the order of application—that is, employ the stronger acid first—or who are content with acid of uniform strength.

* United States Geological Survey. *Bulletin* No. 255. (Series E, Chemistry and Physics, 44).

Incomplete Extraction of Silver.

Not less diverse are the statements as to the completeness of the silver extraction. Campredon puts the average silver retention for bullion assay, tested upon an aggregate of 200 cornets, at 0.0002 grm. According to Beringer, the surcharge may amount to 0.05 per cent ("A Text-book of Assaying, 1890, p. 129). In Brown's "Assaying" (p. 396) it is said that "A small amount of silver will always remain in the cornet, no matter how carefully the manipulations may have been conducted." On the other hand, Ricketts and Miller say that the inaccuracy due to silver retention in parting can be avoided by careful work ("Notes on Assaying," 1897, p. 109). Later, however, they say (p. 128) with respect to gold bullion, "On the other hand, the gold always contains some silver and occluded gases." That cornets retain occluded gases was proved by Graham (*Phil. Trans. Roy. Soc.*, 1866, p. 433), and Varentrapp showed that the amount varies with the temperature of annealing (Rose, "Metallurgy of Gold," p. 481).

Our own experience in the present work is that while sometimes by dissolving a number of beads together traces of silver were detected, quite as often none were found. This was before we learned the need for more than two or three boilings with acid. We believe that some of the unfavourable results reported by various writers may have been due to insufficient treatment with acid and inadequate washing. Not infrequently the direction is given to wash three times with water. That this is insufficient for such cornets as are obtained in bullion assay our own tests have proved to us. (See further).

There is great lack of agreement, too, as to the best gold-silver ratio in the quartation alloy. For instance, Chaudet and Kandelhardt are credited in "Bodeman-Kerl" (Goodyear's translation, 1900, p. 183) with the statement that a gold-silver alloy with 2½ parts of silver retains less silver when parted with nitric acid than an alloy with 3 parts. The same authority attributes to Pettenkofer the statement that the separation can succeed well with as little as 1½ parts of silver to 1 of gold. Rammelsberg (*Ibid.*, p. 184) is quoted to the effect that pure nitric acid dissolves all the silver out of an alloy containing 80 per cent and more of silver, and leaves all the gold behind; and, on the contrary, the separation is imperfect with a proportion of from 15 to 80 per cent of silver.

Impurities in Acid.

Of course, if the acid used for parting contains certain impurities, losses will occur. The impurity most to be guarded against is hydrochloric acid or chlorine. "Bodeman-Kerl" (Goodyear's translation, 1900, p. 187) mention also sulphuric and sulphurous acids. Rose ("Metallurgy of Gold," p. 479) ascribes, on the basis of his own tests, 8 per cent of the total loss in a gold assay to the solvent action of the acid, without specifying further. Lenher (*Journ. Am. Chem. Soc.*, 1904, xxvi., p. 552) confirms the statements of several authors as to the solubility of gold in a hot mixture of strong sulphuric and nitric acids, and that on dilution with water it precipitates, this precipitation being supposedly brought about by nitrous acid formed from the nitric acid. He also shows that the sulphuric-nitric acid mixture dissolves gold even at 0° C.

(Lenher in that paper gives interesting data on the solvent action on gold of a number of bodies other than the halogens, but they are outside the present discussion).

Action of Nitrous Acid.

The statement, which, according to Makins, can be traced to Berzelius, is met with in several places, that loss of gold ensues in parting from the solvent action of nitrous acid; and Makins (*Quart. Journ. Chem. Soc.*, 1861, xiii., p. 99), by repeated boilings of assay gold with nitric acid of specific gravity 1.25 and 1.35, was able to establish a steady loss which he attributed to nitrous acid, though the proof is by no means convincing, as he

does not clearly show the entire absence of traces of hydrochloric acid in his nitric acid, nor of silver in his cornets, nor did he evaporate the filtrates and prove that they contained gold. The active agent could not be nitrous acid formed from the solution of the quartation silver, for its formation must have practically ceased after the second or third boiling. Van Liew obtained astonishing results (*Eng. and Min. Journ.*, lxi., p. 498). He condensed nitrous acid, which he prepared by reducing potassium nitrate with lead and treating the nitrite with sulphuric acid, in a 30 per cent solution of nitric acid in which had been placed leaf-gold. At 15° C. about 1 to 2 per cent of the gold was found to have dissolved, and upward of 38 per cent at 82° C., and 51.9 per cent at an undetermined lower temperature.

In order to obtain light on this point one of us prepared nitrous acid, and tested its effect on spongy gold. Cold aqueous solutions were obtained by the method of Lunge from starch and nitric acid.

Experiment 1.—Twenty-five c.c. of the solution, containing 0.032 grm. HNO₂ and 0.401 grm. HNO₃ per c.c., was placed, in a pressure bottle with very thoroughly washed spongy gold, and left in contact for thirty-six hours, the final temperature being about 30°. Evaporation of the filtrate to dryness showed a small yellowish white residue, which proved to be mostly organic (a blank test gave the same result). When this was heated to redness a faint purple stain remained. This, when dissolved in a few drops of aqua regia and tested with stannous chloride, showed a trace of gold.

Experiment 2.—The solution was more concentrated, holding 0.0548 grm. HNO₂ and 0.075 grm. HNO₃ per c.c. A trace of gold was found.

Experiments 3 and 4.—In each of these 10 c.c. of solution containing 0.062 grm. of HNO₂ and 0.219 grm. of HNO₃ per c.c., was sealed in a tube with spongy gold, and heated for seven hours at about 150°. No gold was found in either case.

It is difficult to see how the above conditions can have differed materially from those in Van Liew's experiments; nevertheless, nitrous acid was made from pure potassium nitrate, and its action on spongy gold observed at a temperature of about 70° to 80° in pressure bottles. No solution of gold could be proved.

In all of the above experiments the acid was filtered through a close plug of ignited asbestos before testing for gold.

The results of these severe tests show that solvent action by nitrous acid during an assay need not be considered. The reported precipitating action of nitrous acid in dilute solution (see above) seems to be in direct opposition to such solvent action. Van Liew's abnormal results must have been due to using a nitrate containing chloride as the source of his nitrous acid.

Action of Nitric Acid.

To test the possible effect of nitric acid itself the following experiments were made:—

Four portions of pure gold were cupelled with three times their weight of silver and 5 grms. of lead each. Five boilings with acid, with intervening washings, were needed to extract the silver to such an extent that an ordinary test failed to show it longer in the extracts. Then No. 1 was boiled with 20 c.c. HNO₃ for fifteen minutes, washed, annealed, and weighed. No. 2 was boiled two periods of fifteen minutes each with acid and properly washed, then annealed, and weighed. With Nos. 3 and 4 the boiling with acid was repeated two and three times respectively, the duration of acid treatment in case of No. 4 lasting between one and two hours. The following table shows that no systematic losses appear, and that in the most severe test no loss whatever occurred. (See table, next column).

The acid thus used was tested for gold as follows:—It was evaporated in clean porcelain dishes, the residues were moistened with a few drops of water and wiped out

	Taken. M.grms.	Found. M.grms.	Loss. M.grm.
1	250.27	249.99	-0.28
2	250.23	250.29	+0.06
3	250.57	250.53	-0.04
4	250.38	250.38	—

clean with small pieces of filter-paper; these were wrapped in lead cornets to which a tiny piece of silver was added, cupelled, and parted.

No. 1 gave no gold. (The absence of gold here shows that the large apparent loss in weight of the cornet, as shown by No. 1 of the table above, was not due to solution of gold).

No. 2 gave 0.01 m.grm. gold.

No. 3 gave 0.01 m.grm. gold.

No. 4 gave no gold.

The acid used for parting was also carefully tested for gold after first precipitating the silver. That from the first four partings combined gave no gold. That from the second gave 0.02 m.grm. gold in all. This gold may have been float gold, though the utmost care was taken to guard against this source of error.

From the above it is plain that the losses in parting with pure acid, whether traces of gold really dissolve or not, are negligible in an ore assay at least.

In conclusion, it may be said that the gold beads, after the repeated treatments with nitric acid detailed above, were dissolved in aqua regia, and the solutions tested for silver with the greatest care, both before removal of the gold and afterwards. In not a single case could the slightest trace of silver be detected.

Summary of Results.

Not all of the conclusions here summarised are to be regarded as fully established, while some are but confirmations of earlier statements by others. More work is needed along certain lines, for there is more than one obscure point connected with the fire assay that needs greater attention than we were able to give it. To what extent the results for cupel losses by different workers will be influenced by a difference in the quality of the cupels used there is, unfortunately, no means of determining. The possibility of such a disturbing influence must always be borne in mind.

It is clearly established that the fire assay by crucible for gold telluride ores gives results which are quite equal to those obtained by the wet way, provided due corrections are made for slag and cupel losses.

The gold loss in the slag is very small, and generally negligible with the charge used by us,* but the cupel losses are very appreciable, and considering the far greater value of gold than of silver, weight for weight, it may be doubted whether the statement that "the loss of gold in the assay of any ore, except when extremely rich, is too small to require correction" (Ricketts and Miller, p. 110) is justified when very exact work is called for. Much depends on what is meant by "extremely rich." Doubtless the ores treated of in Part I. fall within this category, but the losses are there so detectable that correction seems called for with ores far less rich. In view of the very appreciable errors that always occur, and which it is quite impossible to wholly allow for in any ordinary assay, it seems that those who demand an assay balance weighing to less than the hundredth of a milligram are decidedly straining at a gnat if they are willing to swallow the camel of an uncorrected assay.

The cupellation loss of gold by volatilisation is generally small as compared with that by absorption, and at a temperature allowing the formation of abundant feather

litharge is negligible, or perhaps compensated by retention of lead (Table IV.), but the case is otherwise at high temperatures (Tables VIII. and IX.), when it may average one-half of that by absorption in the case of a quartation alloy. The results of Table VI. seem to contradict the first statement, for they show a much greater apparent loss by volatilisation than by absorption.

The loss of gold by absorption is a very important one, and is influenced, far more than is generally supposed, by slight changes in temperature. It is greater with pure gold and alloys poor in silver than with alloys rich in silver.

From a limited number of tests it appears that the ratio of volatilisation loss to absorption loss for quartation alloys at higher than the normal temperature of cupellation is about the same for both gold and silver, or roughly, 30 to 70. This point needs further testing.

Our experiments failed absolutely to show the need for a higher temperature at the end of cupellation with gold beads than with those of silver. The most exact results were obtained when feather litharge was still abundant at the time of brightening.

Furthermore, it is altogether unnecessary to leave gold beads in the muffle for some time after brightening in order to remove the last of the lead, for there is no loss in weight from so doing, but if anything a very slight tendency to increase.

Our results on absorption as influenced by the amount of lead used in cupellation are inconclusive.

The error caused by retention of lead in the beads is one of some magnitude if the results of two careful tests are to be depended on, which showed 0.30 and 0.37 per cent respectively of lead. The amount of this retention is not lessened by leaving the beads in the muffle for some time after brightening.

Silver can be completely extracted from quartation alloys by nitric acid, but more than two repetitions of the acid treatment and subsequent washing are called for if any certainty of complete extraction is to be expected.

Tests made with mixtures of pure nitrous and nitric acids show that the solvent action of the former acid is so slight, if indeed there is any at all, that it need not be considered as a possible disturbing factor in parting.

Similarly, it was shown that the losses in parting with pure nitric acid, whether traces of gold really dissolve or not, are quite negligible, in an ore assay at least.

A SYSTEM OF QUALITATIVE ANALYSIS INCLUDING NEARLY ALL THE METALLIC ELEMENTS.*

By ARTHUR A. NOYES.

INTRODUCTION.

THE aim of this investigation has been to work out in detail a systematic, universally applicable scheme of qualitative analysis which shall include as nearly as practicable all the metallic elements, and which shall make possible their detection even when present in quantity as small as 1 or 2 milligrams.

The great importance, both from a technological and a purely scientific standpoint, which has become attached in recent years to many of the "rare elements"—so-called often only by reason of their traditional and somewhat arbitrary exclusion from the usual schemes of qualitative analysis—has made it highly desirable that a systematic and reliable method for their detection be available. Any chemist who, without previous experience in this field, has occasion to test for small quantities of these elements will infer upon consulting the existing literature, and become

* It is quite conceivable that some ores, differing from ours in the character of their gangue, may give slags that have greater solvent action on gold.

* From the *Technology Quarterly*, xvi., No. 2.

convinced when he begins to experiment, that his problem is beset with difficulties, and will require much time for its solution.

Until within a few years, aside from the brief outlines given in the text-books on qualitative analysis of Rose, Fresenius, Classen, and a few other writers, nothing like a systematic scheme of analysis existed; and it was necessary for the analyst to apply as best he could the isolated separations and tests recorded in such text-books and in the journals. In 1898, Carnot published in his "Traité d'Analyse des Substances Minérales" a scheme of qualitative analysis which included all the more important rare metals. But none of these methods seem to have been subjected to any adequate investigation with reference to the effectiveness of the separations or the reliability and delicacy of the tests; moreover, in a field of work where the closest adherence to the proper conditions is essential to success, the directions given are of a most general character. These schemes, therefore, when tested in the laboratory, are found to be seriously defective, and to have little practical value, except for the detection of large quantities of some of the rare elements. A vast amount of valuable work has, of course been published upon the separation of the rare elements of separate groups, like the rare earths, the platinum metals, and the rarer alkali metals; but this work has been done mainly from the standpoint of quantitative analysis and with reference to the limited number of elements which are found associated in certain important minerals or ores. Of the systematic procedures devised for qualitative purposes ought to be mentioned, however, those of Mylius and Dietz (*Ber.*, 1898, xxi., 3191), and of Leidié and Quennessen (*Bull. Soc. Chim.*, 1902, [3], xxvii., 181) on the platinum metals, and of Possetto (*Chem. Centralb.*, 1898, i., 634) on the rare earths. These quantitative and qualitative investigations have furnished many of the data necessary for the working out of a complete system of qualitative analysis, such as that which forms the object of this investigation.

This work has been in progress for over three years, and has been carried on in large part by research assistants and advanced students, whose valuable aid will be given recognition in connection with the separate groups of metals upon which each worked. The investigation has been liberally assisted by grants from the Warren Fund of the American Academy of Arts and Sciences, and will be published in its final form in the *Proceedings* of that Academy. It has been thought advisable, however, to make in this journal a preliminary publication of the work, as fast as integral portions of it are brought into satisfactory shape, so that the method may be available at once to those who may desire to use it. The work upon it will be continued, in order to test it more thoroughly and to improve it in matters of detail; but it is not likely that the procedure will be radically modified.

The results will be presented in the form that seems best adapted to the purposes of the analyst. The System of Analysis will be primarily divided into a series of Parts, in each of which will be described, as a rule, the detection of the elements precipitated or otherwise separated by a group reagent. Part I. will treat of the Preparation of the Solution; Part II., of the Analysis of the Tungsten and Niobium Groups; Part III., of the Analysis of the Selenium and Silver Groups; Part IV., of the Analysis of the Platinum Group and the Detection of Lead and Tellurium; Part V., of the Analysis of the Ruthenium, Iridium, Copper, and Molybdenum Groups; and the succeeding Parts, of the Rare Earth, Aluminium and Iron, Alkaline Earth, and Alkali Groups. Under each Part is first presented a Tabular Outline which will give a survey of the important steps and the chemical reactions involved in the procedure. This is followed by a General Discussion, in which are presented the reasons for the adoption of the process employed. Then comes the Procedure itself, and the explanatory Notes upon it. Next are presented Confirmatory Experiments and References, which serve to

substantiate the statements made in the Notes, and to justify the details of the Procedure. Finally, are given the Test Analyses, which were made with known mixtures according to the procedure, in order to test its efficiency.

The reader who wishes to get only a general idea of the method and its efficiency will find that the chapters entitled "Tabular Outline," "General Discussion," and "Test Analyses," will suffice for his purpose. The actual analyst will utilise especially those entitled "Tabular Outline" and "Procedure and Notes." Only to investigators in similar lines, or to those who seek the justification of the statements made in the Notes will the chapters on "Confirmatory Experiments and References" be of interest.

The operations are described in the Procedures in much detail; and the reasons for them, the theoretical principles involved, and other incidental matters are presented very fully in the Notes. It may well seem to experienced analysts that the writer has gone to an extreme in this respect; but, in the first place, attention to not obvious details is really essential in many cases in order to secure delicacy of the tests; and, secondly, it has seemed best to make the scheme as effective as possible in the hands of chemists with little experience in this kind of work, even though it involves in some cases rather lengthy descriptions. Even with such a full description, a knowledge of the manipulation of exact qualitative analysis is essential, in order to enable one to detect small quantities of some of the elements. Before analysing an unknown substance, any one using the method for the first time should apply the procedure of each group to a known mixture containing 2 or 3 milligrams of each element.

The directions are almost always given in the form applicable to the case where all elements that could be present are present, the modifications admissible when certain operations lead to negative results being usually sufficiently evident. The scheme is divided into a number of separate "Procedures," a new one being begun whenever the substance has been resolved into two parts (such as a precipitate and filtrate) which are to be submitted to different operations, or whenever two or more collateral procedures converge to a single one. In order to make it possible to see at once the proper sequence of the procedures in an actual analysis, they are numbered consecutively in the description (except that some numbers are omitted at the end of each part to facilitate subsequent revision); and at the beginning of each procedure the one by which the substance was last treated, and at the end of each procedure those by which the two separated portions of it are next to be treated, are referred to by number.

Most of the experiments described under "Confirmatory Experiments and References" are undoubtedly merely confirmations of previously known chemical facts. The application of these facts to the specific conditions of the procedures it was, however, constantly necessary to test; and it has seemed worth while to make the results a matter of record. The experiments are preceded by a reference to the procedure and note to which they relate.

Of the abbreviations employed, only a few need be explained:—G. D. is used for General Discussion; P., for Procedure; N. for Note; C. E. for Confirmatory Experiments; T. A., for Test Analyses. A number within parentheses expresses the specific gravity at 15°; even with diluted acids, the concentration is for brevity usually indicated in this way; thus, HCl (1.02) means an acid made by mixing one volume of HCl (1.12) with five volumes of water. When it is directed to add a variable quantity of a reagent (for example, 5—15 c.c.), the amount used should be adjusted to the size of the precipitate or residue. Certain brief expressions commonly employed in the procedures are fully explained, especially with reference to the manipulative details, under one of the first procedures where they occur; for example, see P. 3, N. 2, as to the exact significance of "evaporate just to dryness," P. 5, N. 6, as to that of "wash the precipitate."

CONDENSATION COMPOUNDS OF CAMPHOR-
OXALIC ACID AND AMINES.*SEVENTH COMMUNICATION ON CAMPHOROXALIC
DERIVATIVES.By J. BISHOP TINGLE, Ph.D., F.C.S.,
and
WILLIAM EDWIN HOFFMAN, Jun., Ph.D.

(Concluded from p. 123).

AMINE DERIVATIVES OF CAMPHOROXALIC ACID (continued).

III. Benzylamine.

1. CAMPHOROXALIC acid (5 grms. = 1 molecule) and benzylamine (4.6 grms. = 2 molecules) were mixed in hot alcoholic solution, and, after evaporating off part of the solvent on the water-bath and cooling, a colourless crystalline mass was gradually deposited. After re-crystallisation from alcohol, it was obtained in the form of distorted rhombohedra, melting at 147.5° with the evolution of a gas. With alcoholic ferric chloride no colour was produced. Analysis showed it to be *benzylamine benzylcamphoformeneaminecarboxylate*.

0.2252 grm. substance gave 15.7 c.c. N at 18° and 770 m.m.

Calculated for C_8H_{14} $\begin{matrix} C : CCOONH_3CH_2C_6H_5 \\ | \\ CO NHCH_2C_6H_5 \end{matrix}$ N. 7.95
Found 8.16

2. When the above compound is heated with sodium hydroxide solution it dissolves, forming sodium benzylcamphoformeneaminecarboxylate. The addition of dilute hydrochloric acid in excess to this solution produces a gummy precipitate, which solidifies after standing in contact with water, and, on re-crystallisation from ethyl acetate forms colourless crystals melting at 140°. In sodium carbonate solution it dissolves with the evolution of carbonic anhydride, and with alcoholic ferric chloride produces no colour. Analysis proves it to be *benzylcamphoformeneaminecarboxylic acid*.

0.2035 grm. substance gave 0.5396 grm. CO₂ and 0.1395 grm. H₂O.

Calculated for C_8H_{14} $\begin{matrix} C : CCOOH \\ | \\ CO NHCH_2C_6H_5 \end{matrix}$ C. 72.52 H. 7.35
Found 72.35 7.6

3. Heated above its melting-point, benzylamine benzylcamphoformeneaminecarboxylate gives off a gas which proved to be carbonic anhydride by the production of barium carbonate when it was passed into a solution of barium hydroxide. The fused material, after the evolution of gas had ceased, was re-crystallised from ethyl acetate, and is deposited in colourless prisms melting at 96.5°. With alcoholic ferric chloride no colour is produced. Analysis showed it to be *benzylcamphoformeneamine*.

0.1885 grm. substance gave 9.8 c.c. N at 16° and 762 m.m.

Calculated for C_8H_{14} $\begin{matrix} C : CH \\ | \\ CO NHCH_2C_6H_5 \end{matrix}$ N. 6.07
Found 5.45

If benzylcamphoformeneaminecarboxylic acid is heated above its melting-point, it gives off carbonic anhydride and forms a compound melting at 96.5°, identical with that formed by heating the benzylamine salt of the acid.

IV. α-Naphthylamine.

1. Camphoroxalic acid (5 grms. = 1 molecule) and α-naphthylamine (6.4 grms. = 2 molecules) were mixed in

PART I.—PREPARATION OF THE SOLUTION (INCLUDING THE DETECTION OF OSMIUM, SILVER, AND LEAD).
Tabular Outline.

Non-metallic Substance.—Heat 1 grm. with HNO ₃ (1.20), dilute and filter. (P. 1).		Metallic Substance.—Heat 0.5 grm. with HNO ₃ (1.20), evaporate, and heat at 120°. (P. 4).	
Residue.—Heat with HF, evaporate with HNO ₃ , and add HNO ₃ (1.05); (if insoluble fluorides remain, digest with SiO ₂ ; evaporate, and heat at 120°. (P. 3).	Solution.—Evaporate, and heat at 120°. (P. 2).	Residue.—Heat with HNO ₃ (1.20), dilute, and filter. (P. 5).	Solution.—Heat with HF, dilute, and filter. (P. 6).
Dehydrated Residue.—Heat with HNO ₃ (1.20), dilute, and filter. (P. 5).	Residue.—Heat with HF, dilute, and filter. (P. 6).	Residue.—Heat with HNO ₃ (1.20), dilute, and filter. (P. 5).	Solution.—Heat with HF, dilute, and filter. (P. 6).
Solution.—All elements except Si, Nb, Ta, W, Sn, Os, and most of the Sb and Ti. (P. 51).	Solution.—Nickelium and tungsten groups (Nb, Ta, Ti, W, Sn, Sb); sometimes also P, As, Bi, Se, Te, V, Mo, Ge. (P. 31).	Residue (MnO ₂ , PbO ₂ , HgS, Au, and the substances named below under "Residues").—Boil with HCl (1.20), then with aqua regia, dilute, and filter. (P. 7).	Solution.—Evaporate, add HCl (1.02), filter. (P. 17).
	Solution.—Chlorides of Pb, Mn, Hg, Au, Pt. (P. 71).	Residue.—AgCl, PbCl ₂ , PbSO ₄ , BaSO ₄ , SrSO ₄ . (P. 18).	Solution.—Fuse with Na ₂ CO ₃ ; heat with water, and filter. (P. 9).
	Solution.—Add HCl and filter. (P. 11).	Residue.—Add HCl and filter. (P. 10).	Solution.—Fuse with K ₂ S ₂ O ₇ . (P. 12).
	Residue.—Fuse with K ₂ S ₂ O ₇ . (P. 12).	Distillate.—OsO ₄ . (P. 14).	Residual Solution.—(P. 15).
	Dark Coloured or Metallic Residue (C, SiC, MoS ₂ , FeCr ₂ O ₄ , Fe ₂ (CN) ₆ , Pt, Ir, Os, Rh, Ru, FeSi ₃ , FeCr ₂ , &c).—Fuse with Na ₂ O ₂ , add water and HCl, and distil into NaOH. (P. 13).		

(To be continued).

* From the American Chemical Journal, xxxiv., No. 3.

alcoholic solution. On evaporating the solvent, greenish yellow crystals were found, which, after purification from alcohol, melt at 165°, with the evolution of gas. With ferric chloride in alcoholic solution no colour is produced. Analysis proved it to be *a-naphthylamine a-naphthyl-tamphoformeneaminecarboxylate*.

0.1532 grm. substance gave 7.5 c.c. N at 16° and 766 m.m.

Calculated for $C_8H_{14} \begin{cases} C : CCOONH_3C_{10}H_7 \\ CO NHC_{10}H_7 \end{cases}$.. 5.66
Found 5.75

2. The above salt dissolves when treated with sodium hydroxide solution, forming sodium *a-naphthylcamphoformeneaminecarboxylate*, which, on acidification with dilute hydrochloric acid, forms a yellow precipitate; this, after crystallisation from benzene, melts at 170°, and is found to be identical with the same compound prepared in another way by Bishop Tingle. It dissolves in sodium carbonate with the evolution of carbonic anhydride, and with alcoholic ferric chloride produces no colour.

3. When either of the above *a-naphthylamine* compounds was heated in order to obtain a camphoformeneamine derivative, a gummy substance was obtained, which could not be induced to crystallise from any solvent.

V. Metatoluidine.

1. Camphoroxalic acid (5 grms. = 1 molecule) and metatoluidine (4.8 grms. = 2 molecules) were allowed to react in alcoholic solution. After evaporation, there were deposited fine needles of a pale lemon colour, which, when re-crystallised from alcohol, melted at 126°; with alcoholic ferric chloride no colour was produced. Analysis showed this substance to be *metatoluidine metatolylcamphoformeneaminecarboxylate*.

0.3153 grm. substance gave 19 c.c. N at 25° and 765 m.m.

Calculated for $C_8H_{14} \begin{cases} C : CCOONH_3C_6H_4CH_3 \\ CO NHC_6H_4CH_3 \end{cases}$.. 6.66
Found 6.78

2. The above salt, when treated with a solution of sodium hydroxide, dissolves, forming sodium *metatolylcamphoformeneaminecarboxylate*, which, on acidification with dilute hydrochloric acid, gives a yellow precipitate. After purification from benzene, crystals are deposited melting at 154°. They dissolve in sodium carbonate solution with the evolution of carbonic anhydride, and with alcoholic ferric chloride give no colour. Analysis shows it to be *metatolylcamphoformeneaminecarboxylic acid*.

0.1497 grm. substance gave 5.65 c.c. N at 10° and 770 m.m.

Calculated for $C_8H_{14} \begin{cases} C : CCOOH \\ CO NHC_6H_4CH_3 \end{cases}$.. 4.47
Found 4.58

3. Both metatolylcamphoformeneaminecarboxylic acid and its metatoluidine salt on heating gave a gum which could not be obtained pure enough for analysis.

VI. Diethylamine.

1. Camphoroxalic acid (5 grms. = 1 molecule) was allowed to react with an alcoholic solution (3.4 grms. = 2 molecules) of diethylamine. A compound crystallising in colourless needles was obtained, which, when purified by re-crystallisation from alcohol, melts at 139.5° with the evolution of a gas. With alcoholic ferric chloride it gives no colour reaction. By analysis the compound is shown to be *diethylamine diethylcamphoformolaminecarboxylate*.

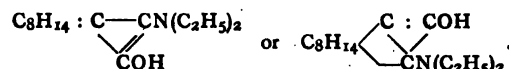
0.1983 grm. substance gives 13.4 c.c. N at 21° and 769 m.m.

Calculated for $C_8H_{14} \begin{cases} CH.CO HCOONH_2(C_2H_5)_2 \\ CO N(C_2H_5)_2 \end{cases}$.. 7.55
Found 7.80

The diethylamine solution used in the preparation of this compound was prepared by dissolving diethylamine hydrochloride in absolute alcohol, adding the required amount of potassium hydroxide, and filtering off the insoluble potassium chloride.

2. When treated with sodium hydroxide solution, the carboxylate just described dissolves, but, on acidification with dilute hydrochloric acid, it is decomposed into camphoroxalic acid and diethylamine. No method of treatment could be devised by which free diethylcamphoformolaminecarboxylic acid could be obtained.

3. When diethylamine diethylcamphoformolaminecarboxylate is heated above its melting-point, diethylamine, water, and carbonic anhydride are given off, and, after purification of the fused material by crystallisation from acetone, the product melts at 153°. With alcoholic ferric chloride the reddish violet colour is produced which is so characteristic of the enol grouping. By analysis it is found to have an empirical formula identical with that of diethylcamphoformeneamine, but, on account of its behaviour towards ferric chloride, its constitution seems, as previously explained (p. 87), to be represented by the formula—



Analysis.—0.2045 grm. substance gave 8.1 c.c. N at 16° and 762 m.m.

Calculated for $C_{15}H_{25}ON$.. 4.56
Found 4.68

VII. Dimethylamine.

1. Camphoroxalic acid (5 grms. = 1 molecule) and dimethylamine (2 grms. = 2 molecules) were allowed to react in alcoholic solution, and, after part of the solvent had evaporated spontaneously, a crystalline compound was obtained. After re-crystallisation from acetone, colourless needles were deposited, melting at 137.5° with the evolution of a gas. With alcoholic ferric chloride it produced no colour reaction. Analysis showed it to be *dimethylamine dimethylcamphoformolaminecarboxylate*.

Analysis.

I. 0.2000 grm. substance gave 0.4452 grm. CO₂ and 0.1661 grm. H₂O.

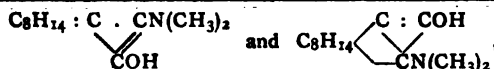
II. 0.2135 grm. substance gave 0.4818 grm. CO₂ and 0.1770 grm. H₂O.

Calculated for—
 $C_8H_{14} \begin{cases} CH.CO HCOONH_2(CH_3)_2 \\ CO N(CH_3)_2 \end{cases}$.. 61.14 9.55

Found { I. 60.71 9.29
II. 61.58 9.27

2. When dimethylamine dimethylcamphoformolaminecarboxylate is treated with sodium hydroxide solution it dissolves, but on acidification with dilute hydrochloric acid decomposes into camphoroxalic acid and dimethylamine.

3. When heated to 140°, dimethylamine dimethylcamphoformolaminecarboxylate gives off water, carbonic anhydride, and dimethylamine, and forms a compound which, when purified by re-crystallisation from acetone, melts at 63°. With alcoholic ferric chloride it gives a purple colour. The alternative formulæ,—



for this compound are discussed on p. 88.

Analysis.—0.2458 grm. substance gave 14.8 c.c. N at 19° and 770 m.m.

Calculated for $\text{C}_{13}\text{H}_{21}\text{ON}$	N.
Found	6.77
	7.01

VIII. Methylamine.

1. Camphoroxalic acid (5 grms. = 1 molecule) and methylamine (2.8 grms. = 2 molecules) were mixed in alcoholic solution. After evaporation of part of the solvent, colourless needles were deposited; they were purified by re-crystallisation from alcohol and melted at 172°. With alcoholic ferric chloride no colour reaction was produced. Analysis proved the compound to be *methylamine methylcamphoformeneaminocarboxylate*.

Analysis.—0.2147 grm. substance gave 20 c.c. N at 14° and 766 m.m.

Calculated for $\text{C}_8\text{H}_{14} \begin{array}{l} \diagup \text{C} : \text{CCOONHCH}_3 \\ \diagdown \text{CO NHCH}_3 \end{array}$	N.
Found	10.51
	11.04

2. When treated with sodium hydroxide, the above salt dissolves, but on acidification with dilute hydrochloric acid decomposes into camphoroxalic acid and methylamine.

3. Upon heating at 175°, methylamine methylcamphoformeneaminocarboxylate gives off methylamine and carbonic anhydride, and forms a compound which, when re-crystallised from ethyl acetate, melts at 131°. With alcoholic ferric chloride, no colour reaction is produced. Analysis proved it to be *methylcamphoformeneamine*.

Analysis.—0.2131 grm. substance gave 13.3 c.c. N. at 11° and 766 m.m.

Calculated for $\text{C}_8\text{H}_{14} \begin{array}{l} \diagup \text{C} : \text{CH} \\ \diagdown \text{CO NHCH}_3 \end{array}$	N.
Found	7.25
	7.50

IX. Benzamidine.

Free benzamidine (2.2 grms. = 1 molecule) was obtained by dissolving the hydrochloride in absolute alcohol, adding the required amount of potassium hydroxide, and filtering off the insoluble potassium chloride. When camphoroxalic acid (5 grms. = 1 molecule) is allowed to react with such an alcoholic solution of benzamidine and the solvent is permitted to evaporate spontaneously, a compound is obtained which may be purified by re-crystallisation from alcohol. It is deposited in colourless prisms; these melt at 184°, evolve a gas, char, and decompose. With alcoholic ferric chloride no colour reaction is produced, and neither acid nor alkali seem to cause any change in the composition of the compound. When heated above its melting-point, it decomposes completely to a carbonaceous mass, from which it is impossible to obtain any definite compound. It did not dissolve in sodium carbonate solution. Analysis proved it to have the empirical formula $\text{C}_{19}\text{H}_{24}\text{O}_4\text{N}_2$. Its probable constitution is discussed on p. 88.

0.1539 grm. substance gave 0.3723 grm. CO_2 and 0.0963 grm. H_2O .

0.2147 grm. substance gave 15.5 c.c. N at 19° and 766 m.m.

	C.	H.	N.
Calculated for $\text{C}_{19}\text{H}_{24}\text{N}_2\text{O}_4$	66.27	6.97	8.14
Found	65.93	7.002	8.30

X. Benzidine.

Camphoroxalic acid (5 grms. = 1 molecule) and benzidine (4 grms. = 1 molecule) were mixed in hot alcoholic solution. When cooled, this deposited a dense mass of

greenish yellow microscopic needles, which, on account of its very sparing solubility in all the ordinary organic media, was extracted with ether to remove any camphoroxalic acid and benzidine. The residue after this treatment formed a fine powder, which gave no colour reaction with alcoholic ferric chloride, and was not affected by acids or alkalis. It melts at 190°, and when heated above this temperature it decomposed to a carbonaceous mass. It is insoluble in sodium carbonate. Analysis proved it to have the empirical formula $\text{C}_{24}\text{H}_{26}\text{O}_3\text{N}_2$; its possible constitution is discussed on p. 88.

0.1489 grm. substance gave 9.4 c.c. N at 21° and 775 m.m.

Calculated for $\text{C}_{24}\text{H}_{26}\text{O}_3\text{N}_2$	N.
Found	7.18
	7.31

XI. Paranitroorthotoluidine.

When camphoroxalic acid (5 grms. = 1 molecule) and nitrotholuidine (6.8 grms. = 2 molecules) are heated at 190° under pressure in alcoholic solution for four hours, a yellow compound is obtained which is purified by re-crystallisation from alcohol. It is deposited in bright yellow needles melting at 192°. This compound is unaffected by acids or alkalis, does not dissolve in sodium carbonate, and produces no colour reaction with ferric chloride in alcoholic solution. Considerable difficulty was experienced in the combustion of the substance for the determination of nitrogen; even when the heating was done most cautiously, explosions occurred, so that the analytical results were unreliable. These explosions were probably due to the presence of the nitro group in the compound. The difficulty was overcome in a thoroughly satisfactory manner by mixing the substance with aluminium powder; under these conditions the nitrogen was evolved during the combustion in a stream which could be easily regulated.

Analysis.—0.2145 grm. substance gave 8.8 c.c. N at 17° and 765 m.m.

Calculated for $\text{C}_8\text{H}_{14} \begin{array}{l} \diagup \text{C} : \text{CH} \\ \diagdown \text{CO NHC}_6\text{H}_3(\text{CH}_3)\text{NO}_2 \end{array}$	N.
Found	4.66
	4.79

XII. Semicarbasine.

Semicarbazine hydrochloride (5 grms. = 2 molecules) was dissolved in as little cold water as possible, and added to an alcoholic solution of potassium acetate (5 grms. = 2 molecules); this solution was then mixed with an alcoholic solution of camphoroxalic acid (5 grms. = 1 molecule). After evaporation of most of the alcohol on the water-bath the solution was greatly diluted with water; a white crystalline precipitate was produced which, on purification by re-crystallisation from alcohol, formed colourless prisms melting at 200°. When dissolved in boiling glacial acetic acid and precipitated by the addition of alcohol, clusters of colourless microscopic needles are deposited; these melt at 209–210°, and correspond to the compound described by Bishop Tingle (*Journ. Am. Chem. Soc.*, 1901, xxiii., 373) as one of two substances formed by the action of semicarbazine on potassium camphoroxalate in alcoholic solution at 100° under pressure. The presence of a second compound, stated by him to be deposited from acetone in colourless needles melting at 218° was not observed; it is apparently not formed under the conditions employed by us. When the compound melting at 209–210°, obtained from acetic acid solution, was dissolved in sodium carbonate solution and precipitated by acids, it formed a jelly, which, after having been dried and re-crystallised from acetic acid, had the same melting-point as before, viz., 209–210°. If the original compound from alcohol, melting at 200°, is re-crystallised from acetone, its melting-point remains constant. With alcoholic ferric chloride, neither substance produces a colour reaction. Both dissolve readily in sodium carbonate, and, as has been stated,

are precipitated by acids from this solution as colourless jellies. Analysis showed that both compounds have the composition of *semicarbasinecamphoformeneaminocarboxylic acid*.

0.2145 grm. substance gave 0.4373 grm. CO₂ and 0.1261 grm. H₂O.

	C.	H.
Calculated for $\text{C}_8\text{H}_{14}\begin{matrix} \text{C} : \text{CCOOH} \\ \text{CO} \text{ NHHNCONH}_2 \end{matrix}$	55.6	6.76
Found	55.5	6.57

The possible constitution and relationship of these compounds has been discussed on p. 102.

XIII. Orthophenylenediamine.

Camphoroxalic acid (5 grms. = 1 molecule) and orthophenylenediamine (2.4 grms. = 1 molecule) were allowed to react in alcoholic solution. The evaporation of the solvent yielded a deposit of fine yellow needles, which melt at 246°. This substance is identical with the compound obtained by J. Bishop Tingle (*Journ. Am. Chem. Soc.*, 1901, xxiii., 375). The proportional quantities of the reacting materials and the conditions of the experiment were varied, but in all cases the same compound was obtained.

The action of camphoroxalic acid on acetylphenylhydrazine, carbamide, paranitraniline, paramineacetophenone, methylaniline, ethylaniline, benzylaniline, ethylenediamine, benzylphenylhydrazine, quinoline, pyridine, dimethylaniline, orthotoluidine, parabromphenylhydrazine, and hydrazine was tried under conditions similar to those already described for other amines, but in no case could a definite compound be obtained. The reaction products were usually gummy substances which could not be obtained in a crystalline form. They were heated for some time at 150–180° in the hope of forming compounds of the camphoformeneamine type, but from these products also no crystalline derivatives could be isolated by the use of the ordinary organic solvents.

Possibly further investigation of the compounds may result in their purification by distillation under very low pressures.

ACTION OF ACYLATING AGENTS ON CAMPHOFORMENEAMINE DERIVATIVES.

Paratolylcamphoformeneamine (3 grms. = 1 molecule) and acetyl chloride (2.6 grms. = 3 molecules) were boiled in solution of anhydrous ether for about six hours; the ether was then evaporated, leaving a compound which crystallised from alcohol in colourless needles melting at 161°. Analysis showed it to be *acetyl paratolylcamphoformeneamine*.

0.1956 grm. substance gave 0.2992 grm. CO₂ and 0.0705 grm. H₂O.
0.5403 grm. substance gave 0.0983 grm. Mg₂P₂O₇ = 0.0747 grm. C₂H₃O.

	C.	H.	CH ₃ CO.
Calculated for— $\text{C}_8\text{H}_{14}\begin{matrix} \text{C} : \text{CH} \\ \text{CO} \text{ N} \text{ C}_6\text{H}_4\text{CH}_3 \\ \text{COCH}_3 \end{matrix}$	77.4	7.74	13.78
Found	77.26	7.47	13.82

Phenylcamphoformeneamine was treated with phenylsulphonic chloride by the Schotten-Baumann method; 1 grm. (= 1 molecule) of phenylcamphoformeneamine was shaken with 1 grm. (= 7 moles) of sodium hydroxide in 10 per cent aqueous solution, and 3.5 grms. (= 5 molecules) of phenylsulphonic chloride, which were added alternately and in small quantities; a compound is finally obtained which by crystallisation from benzene is deposited in stout colourless needles melting at 133°. Analysis shows it to be *phenylcamphoformeneaminophenylsulphone*.

0.2246 grm. substance gave 0.1311 grm. BaSO₄.

	S.
Calculated for $\text{C}_8\text{H}_{14}\begin{matrix} \text{C} : \text{CH} \\ \text{CO} \text{ N} \text{ C}_6\text{H}_5 \\ \text{O}_2\text{SC}_6\text{H}_5 \end{matrix}$	8.10
Found	8.07

β-Naphthylcamphoformeneamine (2 grms. = 1 molecule) and chloracetyl chloride (2.1 grms. = 3 molecules), when boiled for some hours, in anhydrous ether, react to form a bright red substance, which was purified by distillation under diminished pressure. It boiled at 116°, under 45 m.m. pressure and condensed to a colourless crystalline solid, which could be further purified by means of petroleum ether. From this it was deposited in colourless needles melting at 63°. The compound dissolved in sodium-hydrogen carbonate, with evolution of carbonic anhydride, and gave no colour reaction with ferric chloride. It is very unstable, and consequently could not be obtained in a state of purity sufficient to give concordant results on analysis.

The work described in the preceding pages will be continued and extended in various directions. The cost of the greater part of the chemicals which we have used in carrying it out has been defrayed by a grant from the Carnegie Institution; for this we desire to express our thanks and obligations.

PROCEEDINGS OF SOCIETIES.

PHYSICAL SOCIETY.

Ordinary Meeting, March 9th, 1906.

Dr. C. CHREE, Vice-President, in the Chair.

PROF. H. A. WILSON read a paper on "*The Velocities of the Ions of Alkali Salt Vapours at High Temperatures.*"

This paper contains a summary of previous work on the velocities of the ions of alkali salt vapours in flames and gases at high temperatures. It is shown that all the results so far obtained are consistent with the view that any salt of caesium, rubidium, potassium, sodium, or lithium gives in a Bunsen flame negative ions having a velocity of 1000 c.m. per sec. for one volt per c.m., and positive ions having a velocity of about 80 c.m. per sec. This result can be explained by supposing that each salt molecule emits a negative corpuscle which forms the negative ion, and that the rest of the molecule forms the positive ion.

Mr. D. OWEN asked Dr. Wilson how, in reference to the theory that the positive ion is the salt molecule minus an electron, he accounted for the fact, shown in his experiments, that the velocity of these ions was independent of their mass.

Dr. WILSON remarked that the positive ion probably acted as a centre of condensation, and formed an aggregate of molecules whose size depended only on the positive charge.

Dr. J. A. HARKER read a paper "*On some Experiments on Earth-currents at Kew Observatory.*"

Dr. Harker gave an account of experiments made some years ago at Kew Observatory on the earth-currents produced by electric-traction schemes, and on the disturbances they cause on the self-recording magnetic instruments which are kept continuously running there to register the variations in the declination and the horizontal and vertical forces. For these experiments two large earth-plates were buried in the ground about four feet deep and two hundred yards apart. These were connected through a photographic recording voltmeter of high resistance. On the traces given by this instrument the effect of the trains on the Central London Railway was strikingly shown, and specimen records exhibited clearly indicated the timetable of that railway and also any accidental breakdowns.

The nearest point to Kew, namely, Shepherd's Bush, is about six miles distant. The same disturbances, and also those due to special traction experiments carried out on the system of the London United Electric Tramway Company during the period when the Central London Railway was shut down, were also clearly shown on magnetograph curves. The effects are much greater on the vertical force than on the horizontal force or the declination, the latter being only slightly affected. A second system of investigation was to connect the earth-plates through the primary of a transformer, the secondary terminals of which were connected to a sensitive moving-coil galvanometer of suitable period and damping. Under these conditions the galvanometer recorded a ballistic throw for each movement of a tramway controller, while the slower variations due to magnetic storms and other causes were without effect. A telephone similarly connected gave a perceptible sound for each controller movement.

Dr. CHREE expressed his interest in the paper, and remarked that Dr. Harker had shown how magnetic records were disturbed by earth-currents due to electric-traction schemes some years ago. The state of affairs at the present time was very much worse, and the vertical-force records were so disturbed as to be useless. The author's curves showed that there was a quiet time at night, and Dr. Chree suggested this time for the determination of the absolute values of the magnetic elements.

NOTICES OF BOOKS.

Chemistry of the Proteids. By GUSTAV MANN, M.D. (Edin.), B.Sc. (Oxon). London: Macmillan and Co., Ltd. New York: The Macmillan Company. 1906.

THE author of this work had originally intended to prepare a translation of Prof. Otto Cohnheim's "*Chemie der Eiweisskörper*," but finding that it was desirable to extend the scope of the book in some directions, and that the alterations necessarily made in bringing the subject-matter up-to-date were not inconsiderable, he preferred to re-write the book, basing his work upon the second edition of the German, but allowing himself more latitude in some branches, and also not refraining from expressing opinions not always in agreement with those of Prof. Cohnheim. The result has fully justified his decision, and the English work is certainly an advance on the German, not only because of its greater completeness, but also in other respects. It is impossible in a short notice to call attention to all the valuable improvements and alterations which have been made; the subject-matter has been brought as nearly as possible up-to-date; for instance, the synthesis of albumins, as far as known up to September, 1905, is given in full, and Emil Fischer's work on the polypeptides, now being published in the *Berichte der Deutschen Chemischen Gesellschaft* is abstracted up to as recent a date. The biological aspect of chemistry is specially kept before the reader, but the action of ferments has been treated only very shortly—a wise procedure, for a full discussion would have enormously increased the size of the book, and there is no lack of authoritative text-books on the subject.

Conversations on Chemistry. (Part II.). *The Chemistry of the Most Important Elements and Compounds.* By W. OSTWALD, Authorised Translation by STUART K. TURNBULL. New York: John Wiley and Sons. London: Chapman and Hall, Ltd. 1906.

THE second part of these "first steps in chemistry" deals with the most important elements and compounds, and is necessarily more descriptive and somewhat less heuristic. It is again the teacher who will learn most from the book, and any objection that could be brought against the use of the first part by the pupil is increased in Part II., for it is

quite inevitable that the method of question and answer should prove less satisfactory when the learner has obtained some grasp of the first principles and is a little more advanced. The author shows to the highest degree the power of imparting interest to what many generations of learners have regarded as necessary drudgery; even the least original of teachers should be able with very little difficulty to arrange a lecture and experimental work, based upon each chapter of the book, and there could be no comparison between the value of the lessons thus based, and those of the usual type; they would be far easier for the pupil to assimilate, and would do him much more lasting good. In the theoretical parts the author has introduced some slight innovations; thus, he first discusses the neutralisation of acids and bases, and thence deduces the quantitative stoichiometrical laws, of which the beginner is apt to get somewhat hazy ideas when they are presented in the more usual way, deduced from the law of gaseous volumes. So also in the treatment of electrolysis, the atomic theory, and, in fact, all the theoretical parts, great originality is displayed. No teacher of inorganic chemistry should fail to obtain the book; he will find in it much food for thought, and his pupils will undoubtedly derive great benefit from it indirectly.

Lehrbuch der Gerichtlichen Chemie. ("Text-book of Forensic Chemistry"). Vol. II. By Dr. GEORG BAUMERT, Dr. M. DENNSTEDT, and Dr. F. VOIGTLÄNDER. Braunschweig: Friedrich Vieweg und Sohn. 1906.

THIS second edition of Dr. Baumert's valuable book has been thoroughly revised and brought up-to-date by Prof. Dennstedt and Dr. Voigtlander, who, during their long association with the Chemical State Laboratory in Hamburg, must have had opportunities which are probably almost unique of studying the problems of forensic chemistry. This volume deals with the detection of falsifications of writing, chiefly by photographic methods, and the application of biological processes to the identification of different kinds of blood, &c.; the illustrations from the photographs have been carefully prepared, and are most instructive. Much of the information given in the book is quite new, never having been published before, and as it is written in as untechnical language as possible, it will perhaps be found of use to others besides chemical experts. The appendix, on problems connected with incendiaryism, contains much new matter relating to the detection of petroleum, &c.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances de l'Académie des Sciences. Vol. cxlii., No. 8, February 19, 1906.

Ebullition and Distillation of Nickel, Iron, Manganese, Chromium, Molybdenum, Tungsten, and Uranium.—Henri Moissan.—The author finds that the metals of the iron group have very different boiling-points. Manganese is the most volatile of all, and its distillation can be easily effected before that of lime; after this comes nickel, which boils very quietly. Chromium distills regularly under the action of a current of 500 amperes under 110 volts pressure. The ebullition of iron is much more difficult to obtain, and it is preceded by a violent evolution of the gases which this metal dissolves with so much ease. However, by employing more intense currents, and after the first effervescence has calmed down, the ebullition of iron becomes more regular; in twenty minutes, with a current of 1000 amperes under 110 volts pressure, the author distilled 400 grms. of iron. Uranium has a still higher boiling-point; ebullition not being produced until

700 amperes is reached. Molybdenum has not been boiled with a current of 700 amperes. The crystalline powder obtained in all these experiments by condensation of the metallic vapour possesses the same chemical properties as the metal reduced to a fine powder.

Syntheses of Tertiary Alcohols prepared from Paramethylcyclohexane. — Paul Sabatier and A. Mailhe. — Methylcyclohexanone-1, 4, which can easily be prepared from paracresol, reacts energetically on organo-magnesium chlorides, bromides, or iodides, such as RMgBr ; and the action of water on the crystalline mass obtained gives tertiary alcohols of the general formula $\text{CH}_3\text{—CH} < \begin{smallmatrix} \text{CH}_2\text{—CH}_2 \\ \text{CH}_2\text{—CH}_2 \end{smallmatrix} > \text{COH—R}$.

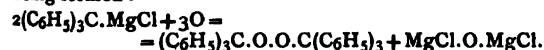
Radio-activity from Springs of Potable Water. — F. Dienert and E. Bouquet. — In order to investigate the natural processes of purification by the soil, and to explain why certain springs emerging from subterranean fissures before filtration do not contain *Bacillus coli communis*, the authors measured the radio-activity of certain springs under the city of Paris.

Condensation of Acetylenic Nitriles with Phenols. General Method of Synthesis of Acrylic β -Oxyphenol- β -substituted Nitriles. — Ch. Moureu and I. Lazennec. — The authors' recent experiments have proved that the acetylenic nitriles, $\text{R—C}\equiv\text{C—CN}$, can be condensed with alcohols, giving addition products which result in the fixation of the alcohols on to the acetylenic liaison. It is known that phenols form with the same nitriles analogous addition compounds. This reaction should be comparable to that of the condensation of the phenols and thiophenols with ethylphenylpropionate and ethyl acetylene dicarbonate, and also to that of the alcohols and phenols with the acetylenic ethers and the acetylenic acetones. In the series of compounds which the authors prepare, a phenolic molecule is fixed on to the acetylenic liaison of the acetylenic nitriles. The hydrolysis of these compounds enables their constitution to be established.

Presence of Formic Aldehyde in Caramelised Substances. — A. Trillat. — The author shows that methylic aldehyde is formed during the incomplete combustion of a number of substances, especially sugar and other substances rich in carbohydrates. He finds that formic aldehyde is found, not only in the gaseous products of these combustions, but also in the residue that is in the caramel, and as a result of a series of experiments finds that sugar at the beginning of caramelisation contains more or less considerable portions of formaldehyde, which influence the original properties. In fact, in sugar refineries it frequently happens that some canes ferment with difficulty, and the formation of methylic aldehyde, owing to the overheating of the cane, offers a very reasonable explanation.

Berichte der Deutschen Chemischen Gesellschaft.
Vol. xxxix., No. 3, 1906.

Magnesium Compound of Triphenylchloromethane. — Julius Schmidlin. — When triphenyl iodo-methane is heated for days with magnesium and benzophenone no magnesium compound is formed, nor when triphenylchloromethane in ethereal solution is heated with pure magnesium; but if a small quantity of iodine is added a reaction at once takes place, and the ether compound of magnesium triphenylchloromethane separates out as a yellow crystalline powder. This magnesium compound could not be analysed because it is very readily oxidised by the oxygen of the air, but the composition appears to correspond to the formula $(\text{C}_6\text{H}_5)_3\text{C.MgCl}$. When oxidised the following reaction takes place, triphenylmethyl peroxide being formed: —



The magnesium compound on addition of water and acids gives a good yield of triphenylchloromethane. By the

action of dry carbon dioxide on the compound triphenyl acetic acid is formed.

Carbon Suboxide (I.). — Otto Diels and Bertram Wolf. — When phosphorus pentoxide acts upon malonic ester, ethylene and a little carbon dioxide are evolved, besides a gas of very piercing odour. The last of these products the authors have succeeded in isolating in the pure state. They found that it is a new oxide of carbon, to which they gave the name carbon suboxide. It is best prepared by using a large excess of phosphorus pentoxide, and allowing the reaction to proceed at 300° ; the products are condensed by means of liquid air, the ethylene being separated by distillation at the temperature of the room. The equation representing the reaction is —



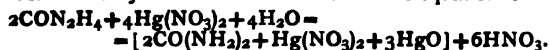
Carbon suboxide is a colourless, refracting, very mobile liquid, possessing a piercing odour, resembling that of acrolein and mustard oil. It attacks the eyes, nose, and respiratory organs, and produces suffocation when breathed in large quantity. It boils at 7° (761 m.m.), but the tension of its vapour is considerable long before the boiling-point is reached. It burns with a smoky blue-edged flame. In all its reactions it behaves like an anhydride of malonic acid. When mixed with water it goes at once into solution, and on evaporating a crystalline mass of malonic acid is left behind. With aniline it yields malonanilide, and it reacts energetically with ammonia, malonamide being formed. With hydrochloric acid it gives malonyl chloride. When it is sealed up in glass tubes and allowed to stand for a day or two at 15° , the liquid slowly turns yellow, and yellow flakes are deposited, which finally form a dark red mass. This mass when rubbed up gives a black powder, the analysis of which gave conflicting results. The preparations thus obtained are soluble in cold water, yielding an intense eosin red colouration. At 37° the decomposition proceeds much more quickly, and at 100° it takes place in a few moments. Elementary analysis showed that the formula of carbon suboxide is C_3O_2 , and when a measured volume was exploded with oxygen the volume of the gases before and after the explosion was the same, as shown by the equation $\text{C}_3\text{O}_2 + 2\text{O}_2 = 3\text{CO}_2$. The vapour density (determined by Hofmann's method) corresponds to the formula C_3O_2 . In composition and properties carbon suboxide recalls the metal carbonyl compounds, especially nickel carbonyl, —



Allotropic Forms of Selenium. — Robert Marc. — The author's experiments, performed with carefully purified selenium, have shown that two very different forms of metallic conducting selenium exist. The grey crystalline form, obtained by heating amorphous selenium, is labile. Denoting this form by A, the author has shown that A goes over into the second form B practically at all temperatures, and with measurable velocity above 170° . This change is accompanied by development of heat. Modification A is a very bad conductor of the electric current at the temperature of the room. On warming, its conductivity rapidly increases, and reaches about twenty times its original value at 170° , and on converting form A into form B the conductivity also increases, being ten times the conductivity of A at 170° ; this increase, however, takes place very slowly. It was to be expected that form B would be the stable form of selenium at all temperatures, but this was not the case. If it was rapidly cooled, the conductivity increased, and at the temperature of the room the conductivity of form B is 1000 to 2000 times greater than that of A. However, when allowed to stand at the temperature of the room the conductivity steadily decreased. It was further found that in the interval 217° to 160° there was equilibrium between the two modifications of selenium, which recalls the analogous case of sulphur for which equilibrium exists between the forms $\text{S}^{\text{A}} \rightleftharpoons \text{S}^{\text{B}}$ between

120–448°; the equilibrium occurs in the liquid phase for sulphur and in the solid for selenium. Amorphous selenium appears distinctly red in thin layers, form A is metallic grey with a faint reddish glimmer, and form B is blue-grey. Amorphous selenium is exceedingly brittle, form A less so, but may easily be powdered, while B is ductile. Neither A nor B is soluble in pure carbon disulphide at the temperature of the room, and the conflicting evidence of other workers upon this and other points is to be ascribed to the impurity of their preparations. Pure selenium shows a very great power of attacking metals, e.g., platinum, and differs in many ways from the impure element.

Quantitative Determination of Urea.—B. Glassmann.—The principle of the method is a modification of Liebig and Pfluger's. First of all, phosphoric acid and sulphuric acid are removed by precipitation with barium nitrate and baryta water; the filtrate is precipitated with nitric acid and silver nitrate, and to the liquid a known excess of titrated mercuric nitrate solution is added, neutralising with sodium carbonate. The precipitate is filtered off, the filtrate acidified with nitric acid, and cold iron alum solution and 30 per cent nitric acid are added till all colouration is removed. The liquid is then titrated with rhodan-ammonium in the usual way till the light brown colour is permanent. Thus the excess of mercury is obtained, and from this may be calculated the urea. The equation is—



This method gives very accurate results, and it has the advantage that the concentration of the urea solution has no influence on the results, as is the case in Liebig's method.

New Series of Ether Complexes of the Magnesium Organic Compounds.—W. Tschelinzeff.—The author has shown that magnesium iodide under ordinary circumstances and in presence of an excess of ether yields a compound of the formula $\text{MgI}_2 \cdot 4(\text{C}_2\text{H}_5)_2\text{O}$. Both analytical and thermic experiments show that when magnesium organic compounds are prepared by Grignard's method, ether complexes containing not one but two molecules of ether are formed. The structure of these compounds is satisfactorily expressed by the formula—



Some other compounds are known in which the halogens appear to be polyvalent.

MEETINGS FOR THE WEEK.

- MONDAY, 26th.**—Society of Arts, 8. (Cantor Lectures). "Fire, Fire Risks, and Fire Extinction," by Vivian B. Lewes.
TUESDAY, 27th.—Royal Institution, 5. (Tyndall Lectures). "The Influence of Geology on Scenery," by John E. Marr, F.R.S., &c.
WEDNESDAY, 28th.—Society of Arts, 8. "Coal Conservation, Power Transmission, and Smoke Prevention," by Arthur J. Martin, M.Inst.C.E.
THURSDAY, 29th.—Royal Institution, 5. "Internal Combustion Engines" (with Experimental Illustrations), by Prof. Bertram Hopkinson, M.A., &c.
FRIDAY, 30th.—Royal Institution, 9. "Recent Progress in Magneto-optics," by Prof. P. Zeeman.
SATURDAY, 31st.—Royal Institution, 3. "The Corpuscular Theory of Matter," by Prof. J. J. Thomson, F.R.S., &c.

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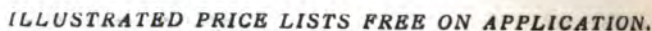
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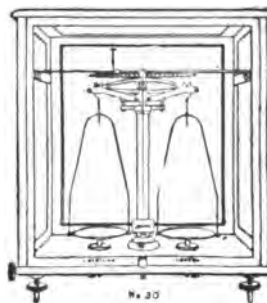
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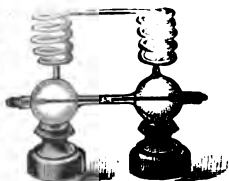
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VOL. XCIII., No. 2418.

ON THE EFFECT OF CALCIUM IN DEVELOPING THE PHOSPHORESCENCE OF SOME RARE EARTHS.

By Sir WILLIAM CROOKES,
Hon. D. Sc. (Oxford, Dublin, and Cape of Good Hope), F.R.S.

ON several occasions arguments have been brought against my views on phosphorescence spectra and their value as tests of elementary bodies. It has been ascertained that, while crude yttria may phosphoresce brilliantly and show the citron (or G δ) line with great brilliancy, highly purified yttria either phosphoresces feebly or not at all. It therefore has been argued that, if a 50 per cent yttrium earth gives the citron line well, and a 100 per cent yttrium earth does not show it at all, therefore the body showing the line resides in the 50 per cent of extraneous matter and not in the 50 per cent of yttria.

The effect of lime and similar bodies in bringing out the phosphorescence of yttria was early forced on my notice. Indeed at first (*loc. cit.*, par. 9) I was inclined to believe that the citron band was actually characteristic of calcium pure and simple, and it was not till I remembered the remarkable result produced in the absorption spectrum of some jargons by the presence of a minute trace of uranium (CHEMICAL NEWS, vol. xix., pp. 121, 142, 205, 277; vol. xx., pp. 7, 104; vol. xxi., p. 73) that I tried the effect of traces of other bodies in educing the citron band.

A systematic series of experiments was made in 1883 (*Phil. Trans. Roy. Soc.*, 1883, Part III., p. 916-917) to ascertain the exact effect of different quantities of lime added to yttria in modifying the phosphorescence spectrum, and the proportions were varied between the limits of of Y/Ca = 1/100 and Y/Ca = 1/1000000. The citron band was visible in all these mixtures, and even when there was a million times as much lime as yttria there was no mistaking its presence, and I said I had no doubt a smaller quantity of yttria could be detected.

In a paper "On the Fractionation of Yttria," read before the British Association in 1886 (CHEMICAL NEWS, vol. liv., p. 155), I said:—"By mixing together yttria and lime . . . the phosphorescing energy of the lime does not spend itself over the whole spectrum, but concentrates itself in greatly reinforcing the yttria bands." Again, speaking of the addition of 10 per cent of lime to alumina containing a trace of yttria, I said:—"The calcium brought out the yttrium band" (*Proc. Roy. Soc.*, vol. xlii., p. 29).

In a paper on "Radiant Matter Spectroscopy," read before the Royal Society in 1887 (*Proc. Roy. Soc.*, xlii., p. 115), I gave a long series of experiments undertaken with a view of ascertaining what was the action of lime on the phosphorescing properties of yttria. I said:—"The effect of calcium on the phosphorescence of yttria and samaria has been frequently referred to in my previous papers; it may save time if I summarise the results here. About 1 per cent of lime added to a badly phosphorescing body containing yttrium or samarium always causes it to phosphoresce well; it diminishes the sharpness of the citron line of G δ , but increases its brightness; it also renders the deep blue line at Ga extremely bright. The green lines of G β are diminished in brightness. Lime also brings out the phosphorescence of samarium, although by itself—or in the presence of a small quantity of yttrium—samarium scarcely phosphoresces at all."

The following extract from my paper on the element samarium (*Phil. Trans. Roy. Soc.*, 1885, Part II., Samarium) illustrates the remarkable effect of lime upon the phosphorescence spectrum:—

"Ultimately two portions of substance were produced—a precipitate (113), containing the supposed new element; and a filtrate, containing the strontium, calcium, and other impurities. Neither the precipitate nor filtrate, tested in the usual manner, showed the orange band anything like so well as the material before such separation, and, indeed, at this stage of the experiments it frequently vanished altogether. Some of the filtrate and precipitate were now mixed together, treated with sulphuric acid, and tested as before; they gave the orange-band spectrum as brightly as did the original substance. The ammonia precipitate might contain any or all of the rare earths. Chemical analysis showed nothing but a calcium salt in the filtrate. Could it be that the union of two bodies was necessary to give this spectrum, and that calcium was one of these?"

"A new factor must now be taken into account—the presence of a calcium compound appears to be necessary. An earthy body which, when treated and tested in the usual manner, fails to show the faintest glow of an orange-band spectrum, can by admixture with calcic sulphate be made to yield a pure and brilliant spectrum."

Besides calcium, many other bodies possess the property of revealing or enhancing the phosphorescence spectrum, and a good many experiments were made upon this point.

"The metals used to mix with it were in the form of sulphates—strontium, barium, glucinum, zirconium, thorium, magnesium, zinc, cadmium, lead, copper, silver, cerium, lanthanum, didymium, aluminium, manganese, tin, bismuth, antimony; also silicic, titanic, tantallic, tungstic, molybdic, and niobic acids. More than half of these bodies possessed the property of conferring 'orange-band' phosphorescence on the precipitate under examination, although by themselves they evinced no power of giving a phosphorescent spectrum."

"Of the great number of bodies used to mix with the earth x (samaria) in these experiments, which acted best?" Ultimately I came to the conclusion that lime, if not the best, was as good as any."

I have also shown that the character of the spectrum produced by a given earth varies very considerably with the particular compound used, the oxides generally giving rise to what I have termed "sharp-line spectra" (*Chem. Soc. Journ.*, Ann. Gen. Meet., March 21, 1889, vol. lv., p. 277); and the sulphates, phosphates, &c., more or less nebulous, shaded bands.

M. Urbain in a communication to the French Academy (*Comptes Rendus*, 1905, vol. cxli., p. 954) suggests that the material giving the group of bands in the ultra-violet spectrum, that I have attributed to an element "victorium," is in reality gadolinium, attempting to overcome the difficulty that this latter earth in its pure state does not show the bands, or at most only shows them very feebly, by suggesting that an "exciting element" is needed to bring out the bands, and the paper in question contains the photograph of a spectrum showing a group of bands very similar in appearance to the victorium group but of slightly different wave-length.

As this difference in the wave-length of a definite group of bands seemed of importance, an attempt was made to get this group together with iron on an instrument of large dispersion; the five-prism quartz spectrograph was used, and an earth very rich in Vc was taken, an exposure of six hours being given; the resulting photograph showed that the main band was double, the centres of its two components being at λ 3116.6 and λ 3119.5, the centre of the blank space between them being at λ 3118.0, and the whole double band extending from wave-length 3115.2 to 3120.7. The Gd-Ca bands of M. Urbain occur between wave-lengths 3136 and 3155.

I have made some experiments to show the effect produced by added lime, both in the form of sulphates and oxides. The details of the experiments are as follows:—

Lime added to M. Urbain's pure europia failed to bring out more than the faintest trace of the Vc bands, but gave

a great concentration of light between wave-lengths 4385 and 3613.

The addition of a small quantity of lime to some pure gadolinia recently received from M. Urbain did not produce any important variation in the phosphorescence spectrum, which was but little different from that given by the pure earth. Another mixture containing 80 per cent of lime with 20 per cent of the pure gadolinia, prepared in the manner described by M. Urbain, viz., by precipitating the mixed earths from their solution as carbonates, and converting them into oxides by ignition, gave only a very faint band in Urbain's new position, λ 3136 to λ 3155.

The phosphorescence spectrum given by the above described mixture containing 80 per cent of lime was again photographed, and then without touching the photographic film the mixed oxides were removed from the vacuum tube, converted into sulphates, returned, and the cathodic spectrum again photographed in such a way that the second spectrum came just below the first.

In the resulting photograph the spectrum given by the oxides consists chiefly of a faint band in Urbain's position, λ 3136 to λ 3155, while that given by the ignited sulphates consisted of bands of the same character and wave-lengths as in the Victoria spectrum.

A mixture was now made of an earth known to be rich in victorium, and pure lime, the latter earth being in about the same proportion as before; this mixture was photographed both as oxide and sulphate on the same film in the way already described; the resulting photograph showed change of character and position in the victorium group in the oxide, and also many other less refrangible lines that are not seen in pure gadolinia.

Further more exact experiments were now made. The victorium earth that was last used was very carefully purified from any lime that might have been present, and a quantity was made into a 10 per cent solution as nitrate.

Some clear crystals of calc-spar were taken, and this also was made into a stock solution containing 10 per cent Ca.

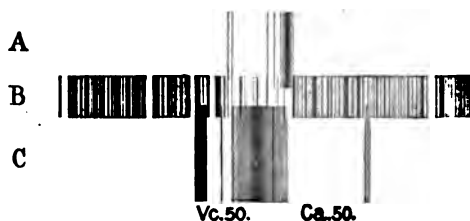
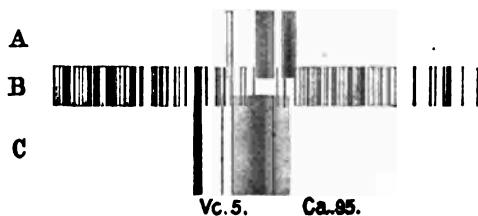
This victorium earth although not pure could have contained little, if any, gadolinium; in the experiments the various proportions of the 10 per cent solutions were mixed together, precipitated by ammonium carbonate, washed, dried, the carbonate calcined at a bright yellow heat, and the cathodic spectrum of the resulting mixed oxides photographed; then, without disturbing the optical arrangements, the oxides were converted into sulphates and their cathodic spectra again photographed, so that the two spectra were one below the other on the same film; the spectrum of iron being interposed as a standard. Three experiments were made with mixtures of Vc + 95 per cent Ca, Vc + 50 per cent Ca, and Vc + 20 per cent Ca. The resulting spectra show that as the proportion of victorium increases, the intensity of the victorium group of bands as given by the sulphates also increases, the mixture of Vc 80 and Ca 20 giving the group in its old character and with considerable intensity; but the most remarkable feature in these compound spectra is that the oxide group of Vc bands is strongest in the mixture containing the smallest proportion of victorium, and they fade out as the proportion of victorium increases, exactly the reverse to the behaviour of the sulphates.

Calcium oxide alone was next tried, and in this case there was a complete absence of the Vc bands, the spectrum giving only a concentration of light in the less refrangible part and a nebulous band in the yellow-green.

Converting this oxide into sulphate changed the cathodic luminescence from golden yellow to bright green, and the photographed spectrum shows a concentration of light in that position.

The victorium earth that has been used in the experiments just described, although rich in Vc, is not by any means so pure as has been obtained from time to time, and in none of these can the percentage of gadolinia as an impurity be at all large. This fact in itself is against the probability of Vc being gadolinia + an exciting agent.

I have many photographs of victorium spectra that show the bands with far greater intensity than I have seen them by using any of M. Urbain's gadolinia earths.



A = Ignited oxide.
B = Iron spark.
C = Ignited sulphate.

M. Urbain's suggestion that Vc is Gd + an exciting agent is improbable, because by no treatment or addition of Ca have I been able to get with pure gadolinia so strong a victorium band as with my old fractions. Moreover, as shown above, the VcCa band is different in character and wave-length from the Vc band.

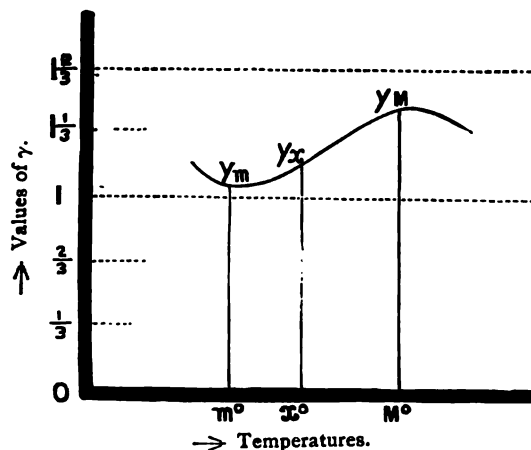
The Chemical Laboratory Fresenius at Wiesbaden.—During the Winter Term, 1905-6, the Chemical Laboratory Fresenius was attended by 47 students. Of these, 35 were from Germany, 4 from England, and 1 each from France, Holland, Hungary, Russia, Spain, Sweden, the United States of America, and Chili. There were three assistants in the Instruction Laboratory and twenty-five in the Versuchsstationen (private laboratories). To the teaching staff of the Institution belong the Directors, Geh. Regierungsrat Prof. Dr. H. Fresenius, Prof. Dr. W. Fresenius, Prof. Dr. E. Hintz, and also Prof. Dr. med. G. Frank, Dr. L. Grünhut, Dr. R. Fresenius, and Architect J. Huber. The Summer Term begins on April 24th. In the Winter Term, 1905-6, besides scientific work, there were many examinations carried on in the different departments of the Laboratory (Versuchsstationen) in the interests of trade, industry, mining, agriculture, hygiene, justice, and government.

QUANTITATIVE RELATION BETWEEN THE SPECIFIC HEATS OF A GAS AND ITS MOLECULAR CONSTITUTION.

By PHILIP BLACKMAN.

FROM the well-known equation $C_p - C_v = R/J$, we obtain $\gamma = 1 + R/JC_v$, where $\gamma = C_p/C_v$.

So long as the molecular structure of a gas remains unchanged during any range of temperature, the value of R also remains constant for that range of temperature. As C_v increases with increase of temperature, it follows that γ will be a continuously diminishing quantity during that interval of temperature (whether this range is measurably large or infinitely small does not, evidently affect the argument). It is an established experimental fact that, the simpler the molecular structure of a gas (i.e., the fewer the number of atoms constituting the molecule), the greater is this value of γ . Hence, if the molecules of a gas begin to dissociate with increase of temperature, we expect that γ will now become continually increasing, till, finally, when the gas is entirely dissociated into simpler molecules, γ will have attained its maximum value for that molecular condition of the gas. If the molecules beyond



that temperature cease to dissociate until a new temperature is reached, γ will once more reach a minimum value, and then go up again to a new maximum so long as the molecules become still simpler in structure. This process would go on repeatedly until the gas molecules attained their simplest structure (i.e., became monatomic, when γ would reach the greatest of all maxima, viz., $1\frac{1}{3}$).

It now remains to show how γ is related to the percentage molecular decomposition of a gas. Let us examine the curve for γ between a minimum value, γ_m (corresponding to a temperature m°), and the next maximum value, γ_M (corresponding to a temperature M°). At the stage represented by γ_m the gas molecules are all alike in structure, at the stage denoted by γ_M the molecules have all been structurally simplified. Therefore the difference between the ordinates at γ_m and γ_M —i.e., $\gamma_M - \gamma_m$ —is equal to 100 per cent decomposition; and any other value, γ_x (at temperature x°), intermediate between γ_m and γ_M , will evidently correspond, proportionately to a percentage decomposition of $100 \frac{\gamma_x - \gamma_m}{\gamma_M - \gamma_m}$.

A similar formula will be applicable to any range of temperature from one minimum value of γ to the next maximum value.

Thus, by plotting or tabulating the values of γ for any gas, from its critical temperature onwards, the results would furnish a valuable means for studying the variations in the molecular complexity of the gas.

A more correct formula is $100 \frac{l_{\gamma x - \gamma_m}}{l_{\gamma_M - \gamma_m}}$ (in which $l_{\gamma x - \gamma_m}$ = length of curve between γ_m and γ_x , and $l_{\gamma_M - \gamma_m}$ = length of curve between γ_m and γ_M).

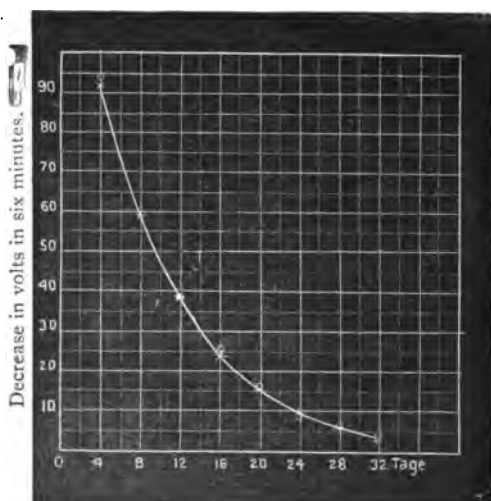
East London Technical College,
London, E.

β-POLONIUM.

By F. GIESEL.

As is well known, the Curies discovered in a bismuth separation from pitchblende the first radio-active substance, polonium, the rays of which have very little penetrating power, and are thus α -rays.

From the beginning of the Curies' investigations I have frequently prepared polonium from uranium earths or various origin, sometimes in relatively large quantity. My polonium, unlike the Curies', when freshly prepared always gave, besides the α -rays, the penetrating β -rays, so power-



o Observed. x Calculated.

fully that the phosphorescence images could be observed very plainly on the Röntgen screen. I then determined the deviability of these rays by the magnet. The direction of the deviation was the same as I had shown for the β -radium rays. However, I had previously proved a difference in the power of penetration of the β -rays of polonium and radium (*Ann. d. Phys. u. Chem.*, 1899, lxi., 93). The image of the hand, for instance, seems to show stronger contrasts with my polonium rays than with radium rays; the former are more easily absorbed. Probably the γ -rays are altogether absent.

Marckwald then showed how a substance emitting only α -rays could be easily separated from the bismuth salts of pitchblende. He thought that his substance was different from the Curies' polonium, and called it radio-tellurium. But it was shown that, both from my polonium and from the Curies' likewise, Marckwald's substance could be prepared by different methods (*Berichte*, 1903, xxxvi., 728). The Curies' polonium, like mine, contained the substance giving α -rays, but mixed in varying (and vanishing) quantities with bismuth and with an allied substance emitting β -rays.

By immersing bismuth or platinum metals in radium solution, I succeeded in reproducing the radiation phenomena of radio-tellurium (*Berichte*, 1903, xxxvi., 2368),

which seemed to confirm my assumption that polonium is bismuth rendered active by radium induction (according to modern conceptions, a decomposition product of radium). Similarly, Rutherford considers that my induced bismuth, like his radium E obtained from radium emanation, is identical with polonium and radio-tellurium. Finally, Mme. Curie (*Comptes Rendus*, Jan., 1906, cxlii., 273) has recently found the same constant of decay to the half value—viz., one hundred and forty days—for her polonium as Marckwald found for radio-tellurium, so that there can no longer be any doubt of the identity of the substance which has been prepared in different ways by Curie, Marckwald, and me from the bismuth salts of pitchblende, and which emits only α -rays.

The substance emitting β -rays, which I may call β -polonium for short, has not been investigated by other workers, probably because it decays too rapidly, so that it can only be found in sufficient quantity in rapidly made preparations. As the determination of the decay constants is of importance for the further characterisation of my substance, β -polonium, I took the opportunity of a recent radium extraction to prepare from the raw barium-lead-radium sulphate, by the old method, a bismuth oxychloride which I used for this purpose.

The observations of the decay of the radiation were made in Elster and Geitel's apparatus at intervals of four days; they are collected in the table given below. The intensity of the radiation is put equal to a decrease of the charge in volts within six minutes, using 0.01 grm.

Time, in days.	Intensity of the radiation.		Δ .
	Observed.	Calculated.	
0	—	144.6	—
4	94.0	92.2	+1.8
8	58.4	58.8	-0.4
12	38.2	37.5	+0.7
16	25.4	23.9	+1.5
20	15.6	15.2	+0.4
24	9.4	9.8	-0.4
28	6.6	6.2	+0.4
32	4.2	3.9	+0.3

(Cf. the graphic representation).

In the determination, the α -radiation of the Curies' polonium contained in the preparation could be neglected, because this can arise only in vanishing amounts from the bismuth salts and the paper envelope used. This was shown in one part of the preparation yielding the above curve, from which the part which gave the α -radiation was removed by immersing a strip of copper in the hydrochloric acid solution. The bismuth oxychloride then suffered no appreciable change in the decay of the β -radiation.

The calculation of the intensity of the β -radiation was performed by putting $I = I_0 e^{-\lambda t}$.

A comparison experiment showed that if t is measured in days the decay constant has the value 0.1128. The constant of decay to the half value is thence calculated to 6.14, and the mean duration of life of an atom yielding the β -radiation to 8.86 days. As the numbers are not to be found among the time constants of the radium derivatives investigated by Rutherford, further experiments are necessary to explain the eventual position of β -polonium in the series of the decay products of radium.—*Berichte*, 1906, xxxix., 780.

Calcium and Strontium Iodomercurates.—A. Duboin.—The author obtains calcium iodomercurate, $\text{CaI}_2 \cdot \text{HgI}_2 \cdot 8\text{H}_2\text{O}$, in transparent needles, which may attain a length of 5 c.m., by slow evaporation in the air of a liquid of the following composition:—Ca, 3.97 per cent; Hg, 21.84 per cent; I, 52.80 per cent; and water, 21.39 per cent. The density of the crystals is 3.25. He also prepares the corresponding strontium salt, $\text{SrI}_2 \cdot \text{HgI}_2 \cdot 8\text{H}_2\text{O}$.—*Comptes Rendus*, cxlii., No. 10.

A SYSTEM OF QUALITATIVE ANALYSIS INCLUDING NEARLY ALL THE METALLIC ELEMENTS.*

PART I. (continued).—PREPARATION OF THE SOLUTION
(INCLUDING THE DETECTION OF OSMIUM, SILVER,
AND LEAD).

By ARTHUR A. NOYES.

(Continued from p. 136).

General Discussion.

MUCH effort has been devoted to determining the best general method for the preparation of a solution of an unknown substance for the analysis for the metallic elements. That some definite method be adopted for this purpose is an essential preliminary to the working out of a general system of analysis, especially when the rare metals are to be included; for the subsequent division into groups must depend to some extent upon the nature of the acids employed as solvents. The aim has been to devise a method of preparing the solution, which in its actual application to substances ordinarily met with shall be as rapid as possible, which shall be so general and so fully developed as to effect the solution of practically all known substances, and which shall adapt itself to sharp and convenient group-separations in the subsequent system of analysis.

In the usual well-known method of procedure with a non-metallic substance, this is heated successively with water, dilute and concentrated hydrochloric acid, nitric acid, and aqua regia. Then the residue, if blowpipe tests show the absence of much reducible metal, is fused in a platinum crucible with sodium carbonate; or, if reducible metal is present, to remove it the residue is first treated with special solvents like potassium cyanide and ammonium acetate, and is then fused in a platinum crucible; or, if the use of such solvents is inconvenient or ineffective, the residue is fused in a porcelain crucible, whereby the constituents of the crucible are introduced. Finally, any residue undecomposed by the carbonate fusion is fused with such special fluxes, potassium disulphate, sodium carbonate and nitrate, sodium carbonate and sulphur, as the apparent nature of the residue suggests. It will be seen that this procedure is very different from the one summarised in the Tabular Outline given above, which has been adopted as a part of the new System of Analysis. The advantages of the proposed modifications should therefore be presented.

An important consideration having a direct bearing on the procedure to be adopted for the preparation of the solution must be first mentioned. This has reference to the fact that certain oxides, namely, those of silicon, tungsten, niobium, and tantalum, do not dissolve, or at any rate do not remain in solution, in the dilute mineral acids commonly employed; so that, when a substance containing these oxides has been brought into solution by an alkaline fusion or treatment with a special acid like concentrated sulphuric or hydrofluoric acid, they separate upon neutralisation of the alkali or upon dilution or replacement of the acid, either immediately or in some subsequent stage of the analysis. This fact has been ignored in the previous schemes of analysis outlined for the rare elements; thus tungsten, in spite of the insolubility of its oxide in acids, is provided for either in the ammonium sulphide solution of the hydrogen sulphide precipitate, or in the filtrate from the ammonium hydroxide and sulphide precipitate; niobium and tantalum, which are bound to precipitate through hydrolysis of their salts when the solution is diluted and heated in the precipitation with hydrogen sulphide, are commonly included among the elements precipitated by ammonium hydroxide. It is therefore essential, unless these elements are to be allowed to appear somewhat

* From the *Technology Quarterly*, xvi., No. 2.

capriciously at different points in the analysis, to remove them at the start. In order to do this completely, however, it is necessary to evaporate the solution to complete dryness, in order to dehydrate their hydroxides and make them completely insoluble in dilute acid. Even though it is true that certain other elements divide themselves between such a residue and the solution, and must be provided for in the subsequent analysis of both portions, this is a far less serious objection than that justified by the failure to provide for the certain detection of tungsten, niobium, and tantalum. It is for this reason that the first series of operations after the decomposition of the substance with strong acids is directed to effecting the removal of the group of elements whose dehydrated hydroxides are insoluble in dilute acid.

The first solvent employed in the proposed method (P. 1) is nitric acid, not hydrochloric acid; the most cogent reason for this being that, if the latter acid were used, certain elements, namely, arsenic, selenium, germanium, mercury, and gallium, would be wholly or partly lost in the subsequent evaporations, owing to the volatility of their chlorides. Hydrochloric acid also has the disadvantage of causing the precipitation of silver, lead, mercurous and thalious chlorides, and thereby making it impossible to determine whether complete decomposition has been effected. Nitric acid, owing to its oxidising power, is, moreover, a more general solvent; but it does on this very account have the disadvantage of oxidising sulphides to sulphates, and causing the separation of the insoluble sulphates of lead, barium, and strontium; and it is to reduce the amount of this oxidation and to make certain crystalline nitrates, like those of lead and barium, more soluble, that a somewhat diluted acid of the specific gravity 1.20 is recommended. Furthermore, nitric acid, unlike hydrochloric acid, leaves all the tin and almost all of the antimony in the insoluble portion of the dehydrated residue.

The first residue insoluble in nitric acid is not treated at once with aqua regia, because the latter solvent decomposes very few substances unacted upon by the former; because the disadvantages of hydrochloric acid just specified apply to its use in some degree; and because any residue undissolved by it, especially if not thoroughly washed, might retain chlorides which would cause the platinum vessel necessarily used in the next treatment with hydrofluoric acid to be attacked.

The residue undissolved by nitric acid is treated with hydrofluoric acid, for the reason that this forms a simple method of dissolving silica and almost all silicates, which otherwise would have to be decomposed by a fusion with sodium carbonate or some other flux. The fusion with sodium carbonate has the disadvantages that the operation is less convenient; that it introduces alkali metals into the solution, which must therefore be tested for in a separate solution prepared in some other way; and that before the fusion can be carried out in a platinum crucible, the residue must be tested for reducible metals, and, if they are present, freed from them, as far as possible, by treatment with special solvents, like hot concentrated hydrochloric acid or ammonium acetate and potassium cyanide solutions. If, however, it is not desired to test for alkali metals, and if reducible metals are probably absent, the residue insoluble in nitric acid (P. 1) may, if the sodium carbonate fusion is preferred to the treatment here described, be treated at once by P. 7 and then by the subsequent procedures.

Both the free and combined hydrofluoric acid must next be completely removed in order to proceed satisfactorily with the analysis; for, otherwise, partial solution of the oxides of the tungsten and niobium groups would take place, and the glass vessels used in subsequent operations would be attacked. Its removal might be easily accomplished by a single evaporation with sulphuric acid to the point of strong fuming, and this would have the advantage of decomposing a few other substances (such as monazite and thorium fluoride) that are not much attacked by

hydrofluoric and nitric acids. A great many experiments were made to test the applicability of this process of decomposition, so much used in quantitative work, to the purposes of qualitative analysis. It was abandoned, however, because the introduction of sulphuric acid complicates the subsequent procedure by preventing the complete separation of the hydroxides of certain elements of the tungsten and niobium groups when the acid is diluted, and by causing the precipitation of some basic sulphates as well as that of the insoluble neutral ones before it is certain that complete decomposition of the substance has been effected. Its abandonment was, moreover, made possible by the discovery of an alternative method of decomposing the insoluble fluorides. It was found, namely, that all of these except thorium fluoride are destroyed by concentrated nitric acid when its action is supplemented by the addition of finely divided silica. This substance evidently acts by combining with the hydrofluoric acid liberated by the strong nitric acid, thus enabling the reaction to become complete in accordance with the Mass Action Law; it was, in fact, this theoretical consideration that suggested its use.

With by far the larger proportion of substances apt to be submitted to analysis, any portion of the dehydrated residue that may be undissolved by dilute nitric acid (P. 5) consists only of silica and oxides of the tungsten and niobium groups, and dissolves completely when it is treated with hydrofluoric acid (P. 6). Still less frequently will there be a residue after the next treatment with hot strong hydrochloric acid and aqua regia (P. 7); for almost all of the fairly common substances previously unacted upon, such as manganese and lead peroxides, mercuric sulphide, silver chloride, lead strontium, and barium sulphates, gold and platinum, are dissolved by these acids. The lengthy description of subsequent procedures, which is added in order to provide for nearly every possible case, must not therefore give the impression that the method here adopted is in its ordinary application long or complicated.

Of the subsequent fusions necessary in special cases, only that with sodium peroxide (P. 13) need receive any general discussion. This flux, suggested by the work of Leidié and Quennessen (*Bull. Soc. Chim.*, 1902, [3], xxvii., 181) was adopted, because it undoubtedly furnishes the best solution of the difficult problem of bringing the rarer platinum metals into a soluble form. The process usually employed for this purpose consists of two parts:—The metal is first brought into a fine state of division by fusing it with a large quantity of lead and treating the fused mass with nitric acid; the residue is then intimately mixed with sodium chloride, and heated to a dark red heat in a current of chlorine. Both parts of this process are extremely tedious and likely to yield incomplete results, even when only half a gram. of metal is to be treated. On the other hand, when the metal is to be fused with sodium peroxide, mechanical processes readily reduce it to a sufficiently fine state of division, and the fusion need not be continued for more than twenty minutes. It is true that the nickel crucible, in which the fusion is best carried out, is much attacked by the flux, but the presence of nickel does not interfere at all with the detection of the platinum metals, or of the other elements which are precipitated by hydrogen sulphide, nor does it interfere very seriously with the detection of the elements present in the precipitate produced by ammonium hydroxide or sulphide, since they can be separated from almost all of the nickel by treating the precipitate with dilute hydrochloric acid. It may be added that before finally adopting this procedure a few experiments were made on metallic iridium with the hope of effecting its solution by heating it to 200° in sealed tubes with bromine or with a mixture of bromine and concentrated hydrobromic acid, but the quantity dissolved was insignificant.

In concluding this chapter it is a pleasure to add that I have been ably assisted in this part of the work by Messrs. J. E. Ober, G. V. Sammet, and W. H. Whitcomb.

Procedure and Notes.

Procedure 1.—If the substance is a metal, treat it by P. 4.

If the substance is not a metal, proceed as follows:—Grind about 1.5 grm. in small portions in an agate mortar until it is so fine that no grit is perceptible when a little of it is rubbed between the fingers. Weigh out roughly about 1 grm. into a casserole, pour over it 10 c.c. HNO_3 (1.20), cover the dish, and digest the mixture on a water-bath for fifteen minutes, or as long as an action appears to be taking place. [If, however, it is thought necessary to test for the presence of osmium in the form of a salt, boil the substance in a distilling flask with HNO_3 , and collect and test the distillate by P. 13 and P. 14]. Then add 20 c.c. boiling water, and allow the residue to settle. [If a light coloured spongy mass of sulphur has separated, remove it by means of a rod or spatula]. Decant the liquid through a filter. If HNO_3 seemed to be slowly attacking the residue, digest it with a fresh portion of HNO_3 , dilute, and decant as before. Wash the residue with hot water twice by decantation, pouring the washings through the filter. Transfer the residue from the casserole, and filter to a platinum dish by rinsing with a little water. Decant or evaporate off the water. (Solution, P. 2; residue, P. 3).

Notes.

1. In order to secure the decomposition of difficultly decomposable substances by the following procedures, it is of fundamental importance that the substance be reduced to a literally impalpable powder, a result that can often be obtained only by the long-continued grinding of the substance in small quantities at a time.

2. One grm. of the substance is usually taken; for, since 1–2 m.grms. of almost any element can be detected by this system of analysis, with 1 grm. the presence of as little as 0.1–0.2 per cent can be determined, which is ordinarily sufficient. It is treated with the acid in a porcelain vessel because of the possible presence of chlorides, which would attack the platinum vessel which has to be used in the subsequent HF treatment (P. 3). The HNO_3 solution is decanted and the residue is washed for this same reason, and also because in many cases the elements which form insoluble fluorides, of which calcium is the most common, are thereby removed, so that the subsequent treatment with SiO_2 (P. 3) becomes unnecessary. The treatment in a distilling flask when osmium can be present as a salt is to avoid the loss of this element by volatilisation, as OsO_4 . See P. 13, N. 9.

3. If HNO_3 attacks the substance with separation of a white precipitate, the latter may consist of hydrated SnO_2 , Sb_2O_3 , SiO_2 , TiO_2 , GeO_2 , Nb_2O_5 , Ta_2O_5 , MoO_3 , or of BaSO_4 , PbSO_4 , SrSO_4 . Certain nitrates, especially $\text{Pb}(\text{NO}_3)_2$ and $\text{Ba}(\text{NO}_3)_2$, may also separate in crystalline form from the strong HNO_3 ; but these dissolve upon adding water and heating to boiling. If a dense yellow decomposition product results, it probably consists of H_2WO_4 . A spongy mass which becomes pasty on boiling the solution is sulphur.

4. If a portion of the substance is not attacked by HNO_3 (1.20), it probably consists of one or more of the following substances:—The oxides and sulphates named in N. 3, the native or ignited oxides of aluminium, chromium, and zirconium, the peroxides of manganese and lead, the silicates and fluosilicates of many elements, sulphide of mercury, fluoride of calcium, the halides of silver, ferrocyanide of iron, carbon, silicon carbide.

Procedure 2.—Evaporate the HNO_2 solution (P. 1) as nearly to dryness as is possible without overheating the residue. Heat in a hot closet at 120° for one hour, or longer if the mass has not then become perfectly dry. (Residue, P. 5).

Note.

1. The evaporation to dryness and heating in the closet at 120° serve to dehydrate and make less soluble in HNO_3 silica and the hydroxides of the elements of

the tungsten and niobium groups, which may at first be dissolved by the HNO_3 . If the dry residue were strongly heated over a flame, mercury compounds might be volatilised, and iron, aluminium, and chromium oxides made insoluble.

Procedure 3.—Add to the residue insoluble in HNO_3 (P. 1) 5–10 c.c. pure concentrated (40 per cent) HF solution, and digest on a water-bath for fifteen minutes. Note whether complete solution takes place. Add 5 c.c. HNO_3 (1.42), and evaporate just to dryness. Cover the residue with HNO_3 (1.42), and again evaporate just to dryness. Add 10 c.c. HNO_3 (1.05), and heat to boiling.

If complete solution took place either on the addition of the HF, or if it occurs on the treatment with the HNO_3 (1.05), evaporate just to dryness a third time; then heat the dish for at least one hour in a hot closet at 120° .

If neither the HF nor the HNO_3 (1.05) produced a perfectly clear solution, evaporate the liquid just to dryness, loosen the particles from the dish, add 10 c.c. HNO_3 (1.42) and 1 grm. pure, ignited precipitated silica, cover the dish, and digest on a water-bath for three-quarters of an hour. Then evaporate the liquid just to dryness, and heat the dish for at least an hour in a hot closet at 120° . (Residue, P. 5).

Notes.

1. The HF used must leave no residue whatever after evaporation and gentle ignition, since otherwise foreign elements would be introduced. It must not contain H_2SO_4 in considerable amount, since otherwise BaSO_4 and PbSO_4 may be left undissolved on the subsequent treatment of the dehydrated residue with HNO_3 (P. 5) and HF (P. 6), and since some of the hydroxides of the niobium and tungsten groups may dissolve. The presence of H_2SO_4 in it may be detected by adding to 3 c.c. of it 10 c.c. HCl (1.03) or 20 c.c. HCl (1.02) and 1 c.c. 10 per cent $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ solution.

2. Whenever, as in the above procedure, it is directed to evaporate a solution "just to dryness," this should be done in such a manner as not to heat any portion of the dried residue much above 100° . This may be accomplished by heating upon a steam-bath, or more rapidly over a small flame, provided proper care be taken; in the latter case after the solution has been reduced to a small volume, the dish must be kept constantly in motion so that the heated parts of the sides as well as the bottom are always wet with liquid, and when nearly dry the dish must be often removed from the flame and shaken in the air, so that the final evaporation will take place at as low a temperature as possible.

3. If the substance is decomposed by HF with the separation of a white precipitate, this indicates the presence of lead, bismuth, lithium, magnesium, or of an element of the alkaline earth or rare earth group. If no such precipitate separates, it shows the absence in quantity as large as 1 m.grm. in the part of the original substance insoluble in HNO_3 , of lead, bismuth, calcium, and all the elements of the rare earth group. Since LiF , MgF_2 , SrF_2 , and BaF_2 are somewhat soluble, these precipitates separate only in the presence of considerable quantities of the elements—in the presence of, roughly, 5 m.grms. Li, 2 m.grms. Mg, 8 m.grms. Sr, 30 m.grms. Na, when 5 c.c. HF are used.

4. The HF solution must not be evaporated to dryness without the addition of HNO_3 ; for much titanium and tantalum would then volatilise as TiF_4 and TaF_5 . In the presence of HNO_3 , however, the loss of these elements is inconsiderable, undoubtedly owing to the fact that this acid decomposes their fluorides. In the evaporation with HF and HNO_3 there is no loss of mercury, arsenic, selenium, germanium, or antimony—the elements which volatilise to some extent out of a boiling concentrated HCl solution.

5. If complete solution takes place on the addition of HF, the three evaporations with HNO_3 remove the HF completely, even in the presence of the elements

titanium, niobium, tantalum, which have a great tendency to form complex fluorine containing acids. In that case, the treatment with SiO_2 can be dispensed with; and this is desirable, whenever practicable, both because it saves time at this point and because it makes it possible to determine immediately upon the treatment of the dehydrated residue with HNO_3 whether or not elements of the tungsten and niobium groups are present.

6. In case the HF precipitate consists of even large quantities (500 m.grms.) of PbF_2 , BiF_3 , LiF , MgF_2 , SrF_2 , or BaF_2 , or of a small quantity (less than 50 m.grms.) of CaF_2 , the two evaporations decompose it completely, so that these elements pass into solution on adding dilute HNO_3 , and the addition of SiO_2 is unnecessary.

7. If oxides of the tungsten and niobium groups are alone present in the first residue, these (with the possible exception of SnO_2) will yield a clear solution upon the treatment with HF (see P. 6, N. 1), but will again separate after the evaporations with HNO_3 ; in this case, in which HF, but not HNO_3 , yields a clear solution, it is, of course, also unnecessary to treat with SiO_2 .

8. When, however, CaF_2 or rare earth fluorides separate in considerable quantity on the addition of HF, it is not practicable to decompose them by repeated evaporations with HNO_3 alone. But the addition of SiO_2 and digestion with it and strong HNO_3 effect complete decomposition of all the fluorides (except ThF_4 , which may remain partially undecomposed as a dense crystalline powder). The SiO_2 combines with the HF that is liberated by the HNO_3 , and thus, in accordance with the Mass Action Law, enables the decomposition to become complete.

9. CaF_2 , even when present in considerable quantity after the two evaporations with HNO_3 , may yield a homogeneous, slightly milky colloidal solution upon the addition of dilute HNO_3 . This must not be mistaken for a true solution, the production of which alone warrants the omission of the SiO_2 treatment.

10. The SiO_2 used must leave no residue whatever upon treatment with HF and evaporation of the solution. It may be prepared by diluting commercial water-glass with three times its volume of water, adding HCl as long as precipitation continues, decanting, boiling the precipitate two or three times with water, drying it in a hot closet, heating it two or three times with HCl (1:20), washing it till free from acid, drying it in a hot closet, and finally igniting it.

11. The heating at 120° is necessary in order to make silica and the oxides of tin, tungsten, and tantalum entirely insoluble in HNO_3 . This is even more necessary after they have been dissolved by HF than when treated with HNO_3 alone. If the residue were overheated during the evaporations, other oxides, like those of iron, aluminium, and chromium, might not dissolve completely on the subsequent treatment with HNO_3 .

Procedure 4.—If the original substance is a metal, convert it into a form that offers as much surface as possible by grinding it in an agate mortar, hammering it in a "diamond" steel mortar, filing it with a fine steel file, shaving it with a knife, or converting it into borings with a drill. Weigh out roughly about 0.5 grm. into a casserole, pour over it, very gradually if a violent action occurs, 10 c.c. HNO_3 (1:20), cover the dish, and heat the mixture on the water-bath as long as any evolution of nitrous fumes occurs, adding more acid if the action is renewed thereby, or a little water if crystalline salts have separated. Then evaporate the liquid just to dryness, and heat the residue in a hot closet at 120° for an hour, or longer if the mass has not then become perfectly dry. (Residue, P. 5).

Note.

1. See P. 1, N. 3 in regard to precipitates that may separate during the HNO_3 treatment, and P. 3, N. 11 regard to the dehydration of the residue.

(To be continued).

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, March 15th, 1906.

Prof. R. MELDOLA, F.R.S., President, in the Chair.

MESSRS. S. L. Courtauld, L. H. Durrans, and R. W. L. Clarke were formally admitted Fellows of the Society.

Certificates were read for the first time in favour of Messrs. Ernest Barrett, B.Sc., 56, Elswick Road, Lewis- ham, S.E.; Percy Garratt Chamberlain, M.A., 3, Market Place, Rugby; Cecil Wyatt-Edgell, B.A., Cowrey Place, Exeter; O. Bertram Foy, 16, Burlington Road, Dublin; John Adam Watson, 8, Powis Gardens, Notting Hill, W.

A certificate has been authorised by the Council for presentation for ballot under By-law I. (3) in favour of Ram Chandra Mukerjee, B.A., of Jaipur, Rajputana, India.

The PRESIDENT announced that Dr. G. T. Morgan had found it necessary to resign the Editorship of the Society's publications, and the Council had appointed Dr. J. C. Cain to succeed him.

Attention was drawn to the fact that the Sixth International Congress of Applied Chemistry will be held in Rome, beginning on April 25th, 1906.

Of the following papers, those marked * were read:—

*53. "*The Interaction of well-dried Mixtures of Hydrocarbons and Oxygen.*" By WILLIAM ARTHUR BONE and GEORGE WILLIAM ANDREW.

The results of experiments carried out chiefly with mixtures of ethylene and oxygen, well dried by previous long contact with re-distilled phosphoric oxide, do not support the view that the presence of steam is essential to the combustion of a hydrocarbon. It was shown that an amount of desiccation which almost inhibits the combination of hydrogen and oxygen (electrolytic gas) at 525° does not appreciably retard the oxidation of ethylene. The authors therefore conclude that oxygen is directly active in hydrocarbon combustion.

*54. "*The Explosive Combustion of Hydrocarbons.*" By WILLIAM ARTHUR BONE and JULIEN DRUGMAN.

The authors showed, as the result of an exhaustive study of the explosive combustion of a number of different gaseous hydrocarbons, including paraffins up to butane and olefines such as ethylene, propylene, and the butylenes, that the theory of the preferential combustion of carbon completely breaks down when applied to hydrocarbon flames. The results of the research constitute a strong argument in favour of the view that there is no essential difference between the slow and rapid combustion of a hydrocarbon, and that explosive combustion probably involves the initial formation of unstable hydroxylated molecules, which subsequently undergo thermal decomposition into simpler products. Very striking differences as regards the composition of the end-products are obtained when explosive combustion occurs in the system $\text{C}_x\text{H}_y + x/2\text{O}_2$, according as the hydrocarbon is a paraffin or an olefine. In the case of a paraffin, there is always a separation of carbon and a large formation of steam, whereas in the case of the corresponding olefine there is no separation of carbon, and the cooled products consist almost entirely of carbon monoxide and hydrogen in accordance with the empirical equation $\text{C}_n\text{H}_{2n} + n/2\text{O}_2 = n\text{CO} + n\text{H}_2$. In the case of an olefine, it was shown that on reducing the proportion of oxygen much below that indicated by the expression $\text{C}_n\text{H}_{2n} + n/2\text{O}_2$, not only does carbon separate in the flame, but also a large formation of steam occurs, a result which is quite incompatible with the theory of the preferential combustion of carbon in hydrocarbon flames.

Comparative experiments on the explosion of mixtures corresponding to $\text{C}_2\text{H}_6 + \text{O}_2$, $\text{C}_2\text{H}_4 + \text{H}_2 + \text{O}_2$, and $\text{C}_2\text{H}_2 + 2\text{H}_2 + \text{O}_2$ respectively prove that the process cannot

be regarded as involving the primary dissolution of the hydrocarbon, followed by a distribution of the oxygen between free carbon and hydrogen.

Incidentally, it was shown that the affinity of hydrocarbons for oxygen at high temperatures is enormously greater than that of hydrogen, so that whenever mixtures of hydrocarbons and hydrogen react with a supply of oxygen less than is required to complete the initial combustion stages of the hydrocarbon, the latter is burnt almost to the entire exclusion of the hydrogen. Hence, in hydrocarbon flames, the initial stages of the oxidation of the hydrocarbon probably take precedence of all other chemical processes.

DISCUSSION.

Prof. SMITHELLS said that he was very ready to confess any mistake he might have made in giving currency to the idea of a "preferential oxidation" of carbon in the combustion of hydrocarbons. He admitted at once that the expression was not universally applicable, and he would endeavour in future not to use it without the necessary qualifications. He wished, however, to remind Fellows what the history of the matter really was. When he began to experiment with flames, he was under the conviction, which he believed was shared by chemists in general, that when a hydrocarbon was burned with a restricted supply of oxygen, carbon rather than hydrogen would remain unoxidised. That view lay at the root of the explanation of the luminosity of hydrocarbon flames then current. When, experimenting with the aerated flame of ethylene, he had found that the interconal gases contained a large quantity of free hydrogen and no unoxidised carbon, and when he had found this to be true of hydrocarbon flames in general, he felt that a complete revolution must be made in our views, and it was then that he used the expression preferential combustion of carbon. In describing chemical changes, it was customary to name the substances used and the substances ultimately obtained. Whatever transitory products might be formed, however the mechanism might be regarded, it was the custom to say, for example, that zinc and strong sulphuric acid give sulphur dioxide, zinc sulphate, and water. We might suppose that zinc first displaced hydrogen from the acid, and that the hydrogen reduced some more of the acid, or that the zinc was oxidised by the acid and the oxide then dissolved in more acid. Then, again, hydrogen sulphide and free sulphur were to some extent formed. In such an apparently simple reaction as this, we could affirm nothing with certainty about the actual mechanism. Therefore, we said simply that zinc and sulphuric acid give sulphur dioxide, water, and zinc sulphate. In like manner, it might be said that in the case of hydrocarbon flames burning with a restricted supply of oxygen the carbon was preferentially oxidised.

With regard to the probable course of the oxidation, he considered that Dr. Bone had made out a very strong case. It was undoubtedly possible that all the stages which the author had indicated might be passed through; he did not think the explanation presented great difficulties from the standpoint of the kinetic theory of gases. On the other hand, it was undoubtedly possible that the final condition of equilibrium might be reached in one bound, just as a stone under impulse might reach the foot of a hill in one bound instead of following the contour of the slope. It was a question of the balance of evidence. The most serious piece of experimental evidence he knew against Dr. Bone's view was, that in those parts of a flame where the partial combustion of hydrocarbons is taking place there is to be seen a spectrum which he believed to be only known where carbon and oxygen were uniting to form carbon monoxide. That spoke for a direct attack of the oxygen on the carbon. The solution of the question was one of extreme difficulty, and, as he had said, it involved the careful weighing of circumstantial evidence.

Having dealt, Dr. ARMSTRONG said, with Dr. Bone's work elsewhere, and being in agreement with him on the main issue, there were but few matters to which he need

refer. One of the most striking points brought before them was the incombustibility of hydrogen as compared with hydrocarbons—he thought that it would have to be recognised that hydrogen was not the active, attractive element it was commonly supposed to be in hydrogen compounds, but that more often than not it simply became pushed aside, as it were. The carbon appeared to be primarily the attractive element, and in this restricted sense Prof. SmitHELLS was justified in speaking of the preferential combustion of the carbon in hydrocarbons. As to the influence of moisture on the combustion of hydrocarbons, it was to be expected that a hydrocarbon-oxygen mixture would be much more sensitive to change than one of hydrogen and oxygen. Such questions *must* be regarded from the point of view of some consistent theory of chemical change; it would be very difficult to obtain direct experimental proof that conducting water was essential to the occurrence of the combustion. The view he had expressed twenty years ago in that room, that a mixture of hydrogen and oxygen alone would be found to be incombustible, was now accepted, however, and he did not hesitate to assert that it was necessary to extend the argument from hydrogen to hydrocarbons, but would not be surprised if another twenty years passed before it were recognised that a mixture consisting simply of a hydrocarbon and oxygen was incombustible.

Dr. BONE, in reply, remarked that the discussion had not elicited any definite criticism of the views he had advanced in the paper. His own position was simply this. In conjunction with Dr. Drugman, he had discovered certain facts which proved that preferential combustion of carbon did not take place in hydrocarbon flames, but which could be perfectly well explained on the supposition that hydroxylated or oxygenated molecules were initially formed. He had endeavoured to form a mental picture of the processes going on in the flame, but, beyond expressing the conviction that the oxygen is in some way actually incorporated with the hydrocarbon before the system breaks down into simpler products he did not wish to put forward any theory otherwise than as a working hypothesis.

*55. "The Occurrence of Methane amongst the Decomposition Products of certain Nitrogenous Bases as a Source of Error in the Estimation of Nitrogen by the Absolute Method." By PAUL HAAS.

The gas collected over caustic potash in a Schiff's nitrometer during the estimation of nitrogen by the absolute method was found in the case of more than twelve different compounds to contain methane. The accumulation of this gas was prevented by replacing the copper oxide ordinarily used in such analyses by lead chromate, which effectually oxidises any methane formed.

DISCUSSION.

Mr. CARR remarked that the different results obtained when the base or its hydrochloride are burnt and the inhibitive influence of cuprous chloride confirmed the results obtained by Prof. Dunstan and himself. These experiments had shown that methane can only be burnt with great difficulty by passing it over strongly heated copper oxide. From the evidence now adduced he thought that the use of cuprous chloride in determinations of nitrogen by the absolute method should become general. More methane is obtained by the "carbon dioxide" method than by Frankland and Armstrong's vacuum method.

Mr. W. A. DAVIS stated that in 1896, shortly after the publication of Prof. Dunstan and Mr. Carr's results, he had found that 1-nitro-2-methoxynaphthalene gave abnormally high results for nitrogen estimated by the absolute method, and that the "nitrogen" collected was inflammable. The numbers obtained with the substance agree closely with those required for a dinitro-derivative, but the fact that normal results were obtained with the derived 1-amino- and 1-acetylamino-2-methoxynaphthalene clearly established the true nature of the compound. It was doubtful whether the formation of methane could be attributed to any special structure, but it seemed, under

exceptional conditions, to be associated with the presence of a methyl or methoxyl group.

Dr. BONE said that with regard to combustions over a hot surface of copper oxide, he had found that the rate of disappearance of hydrogen when electrolytic gas $2\text{H}_2 + \text{O}_2$ was circulated over copper oxide at 200° was only about 1/30th of the corresponding rate when a mixture of $2\text{H}_2 + \text{N}_2$ was employed. The oxygen in the electrolytic gas was condensed on the hot surface, forming a film which actually protected the copper oxide from the reducing action of hydrogen. Possibly other gases would be similarly condensed on the surface. He was not at all sure that the usual practice of using oxygen instead of air in ordinary combustion analyses over copper oxide was a good one.

56. "Studies on Comparative Cryoscopy. Part IV. The Hydrocarbons and their Halogen Derivatives in Phenol Solution." By PHILIP WILFRED ROBERTSON.

The hydrocarbons and their halogen derivatives are characterised by the fact that they give low molecular depressions, which decrease slowly with the concentration. This phenomenon appears to be intimately connected with the association of the solvent, as it is not exhibited to the same extent by the least associated substituted phenols, *o*-cresol, thymol, guaiacol, and *o*-nitrophenol.

It is also shown that benzene does not differ in behaviour from the other hydrocarbons examined, and hence its low molecular depression cannot be attributed to the formation of a solid solution, as Bruni endeavours to prove (*Gazzetta*, 1898, xxviii., [i.], 249). By employing van Bijlerts' method, his results show that the benzene forms a 30 per cent solid solution in the phenol. From this it is concluded that van Bijlerts' method is not always trustworthy, and, moreover, it is not justifiable to assume the existence of a solid solution when the observed molecular depression differs only slightly from the theoretical.

57. "The Displacement of Acid Ions." (Part I.). By ALFRED FRANCIS JOSEPH.

The author described his investigations on the quantitative action of hydrochloric acid on the nitrates of potassium, sodium, and strontium, and of nitric acid on the corresponding chlorides. An equivalent of the salt in aqueous solution is mixed with x equivalents of the acid, the mixture evaporated to dryness on the water-bath, and the proportion of chloride in the residue determined by titration with standard silver nitrate solution.

If the quantity of salt transformed be y , then it is found that the equation $y = a \log_{10} x + b$ holds good over a considerable range of values of x , a and b being constants depending on the particular acid and salt used. These constants increase and decrease simultaneously with the ratio of the solubilities of the two salts concerned in the transformation. It was found that to transform half the nitrate into chloride by one evaporation with hydrochloric acid requires about 1.5 equivalents of acid in the case of sodium nitrate, 2.9 for potassium nitrate, and 12 for strontium nitrate.

On the other hand, to transform nine-tenths of the chloride into nitrate requires 1.6 equivalents of nitric acid in the case of sodium chloride, 1.4 for potassium chloride, and 1.05 for strontium chloride.

58. "Additive Compounds of Arylamines with Aromatic Nitro-derivatives." By CHARLES LORING JACKSON and LATHAM CLARKE.

NOTE.—The investigations on additive compounds of trinitrobenzene with amines formerly described (*Ber.*, 1904, xxxvii., 176), and in this paper were undertaken before a copy of J. J. Sudborough's second paper (*Trans.*, 1903, lxxxiii., 1334) had been received by the authors, who thereupon discontinued their experiments.

The following additive compounds have been prepared by the authors in addition to those already described (*Ber.*, 1904, xxxvii., 176).

4 : 6 - *Dibromo-1 : 3 - dinitrobenzene dimethylaniline*, $2\text{C}_6\text{H}_2\text{Br}_2(\text{NO}_2)_2 \cdot \text{C}_6\text{H}_3\text{NMe}_2$, which is precipitated as a

heavy red oil. When alcohol is added to a solution of the nitro-compound in an excess of dimethylaniline, it quickly solidifies to short deep red prisms melting at 50° .

0.3487 gave 0.3373 AgBr. Br = 41.12.

0.1762 ,, 0.1705 AgBr. Br = 41.16.

$\text{C}_{20}\text{H}_{15}\text{O}_8\text{N}_5\text{Br}_4$ requires 41.39 per cent.

The compound is extremely unstable, and readily loses dimethylaniline when exposed to the air.

4-*Chloro-1 : 3 : 5-tribromo-2 : 6-dinitrobenzene dimethylaniline*, $\text{C}_6\text{ClBr}_3(\text{NO}_2)_2 \cdot 2\text{C}_6\text{H}_3\text{NMe}_2$, obtained in the same way as the preceding compound, crystallises in slender yellow needles, and melts at 105° .

0.086 gave 0.0889 AgCl and AgBr; Cl + Br = 40.25.

$\text{C}_{20}\text{H}_{11}\text{O}_8\text{N}_5\text{Cl}_2\text{Br}_6$ requires Cl + Br = 40.43 per cent.

The following compounds give red colourations with dimethylaniline; in certain cases the colour is removed by the addition of solvents, and in no case has a definite crystalline additive compound been isolated. Nitrobenzene, *a*-nitronaphthalene, *o*-nitrophenol, *m*-dinitrobenzene, 4-bromo-1 : 3-dinitrobenzene, 4 : 6-di-iodo-1 : 3-dinitrobenzene, 1 : 3 : 5-trichloro-2 : 4-dinitrobenzene, 4-bromo-3 : 5-dinitrobenzoic acid (which yields a salt melting at $120-125^\circ$), 4-ethoxy-3 : 5-dinitrobenzoic acid, 4-*iso*-amylxy-3 : 5-dinitrobenzoic acid, and picric acid.

Methylaniline and *s*-trichlorotrinitrobenzene yield an unstable additive compound, which crystallises in red plates, melting at about 78° ; it becomes colourless when exposed to the air or when washed with ether, chloroform, or acids.

Several of the compounds of toluidines with trinitrobenzene and trinitrotoluene have been prepared (compare, Hepp, *Annalen*, 1882, ccxv., 344; Noetting and Sommerhoff, *Ber.*, 1906, xxxix., 76). The compounds with trinitrotoluene are much less stable than those with trinitrobenzene.

The additive compound of *p*-toluidine and trinitrotoluene, which Sommerhoff could not obtain, may be prepared by fusing a mixture of the two components, adding a very small amount of toluene, and allowing to cool; it crystallises in dark red needles melting at about 68° .

0.2845 gave 41.4 c.c. of moist nitrogen at 20° and 768 mm. N = 16.82.

$\text{C}_{14}\text{H}_{14}\text{O}_6\text{N}_4$ requires N = 16.76 per cent.

s-Trichlorotrinitrobenzene and pyridine form an extremely unstable additive compound, $\text{C}_6\text{Cl}_3(\text{NO}_2)_3 \cdot \text{C}_5\text{H}_5\text{N}$, which crystallises in slender red crystals.

0.1232 gave 0.1224 AgCl. Cl = 24.56. $\text{C}_{11}\text{H}_5\text{O}_6\text{N}_4\text{Cl}_3$ requires 26.87 per cent.

When kept for a short time, this substance sets to a black resin.

59. "Influence of Substituents in the Trinitrobenzene Molecule on the Formation of Additive Compounds with Arylamines." By JOHN JOSEPH SUDBOROUGH and NORMAN PICTON.

The formation of additive compounds between *a*- or *β* -naphthylamine and *s*-trinitrobenzene derivatives is completely inhibited by the introduction of three methyl-, two methoxy-, or three bromo-radicles into the trinitrobenzene molecule. *s*-Trichlorotrinitrobenzene, however, is still capable of combining with *a*-naphthylamine. Various additive compounds are described.

The compounds derived from the naphthylamines and chloro-derivatives of the di- and tri-nitrobenzenes readily lose hydrogen chloride, yielding condensation products of the type of picryl- *β* -naphthylamine, $\text{C}_6\text{H}_2(\text{NO}_2)_3 \cdot \text{NH} \cdot \text{C}_{10}\text{H}_7$. Several compounds of this type have been examined, and the following points established:—1. Most of the compounds exist in two distinct modifications, namely, a sulphur-yellow and a bright red variety. These may be mutually transformed one into the other by simple methods. 2. They form mono-potassium salts which are readily hydrolysed by water. 3. They do not yield acetyl deriva-

tives. 4. Some form extremely unstable additive compounds with arylamines. 5. It has not been found possible to replace the one arylamino-group, $\cdot\text{NH}\cdot\text{C}_6\text{H}_5$, for example, by another, such as $\cdot\text{NH}\cdot\text{C}_{10}\text{H}_7$.

60. "The Relations between Absorption Spectra and Chemical Constitution. Part IV. The Reactivity of the Substituted Quinones." By ALFRED WALTER STEWART and EDWARD CHARLES CYRIL BALY.

The reactive power of carbonyl groups in quinones has been shown by Kehrman (*Ber.*, 1888, xxi., 3315; *Journ. Prakt. Chem.*, 1889, [2], xxxix., 399; xl., 257) to be greatly influenced by the replacement of the hydrogen atoms of the quinone nucleus by methyl radicles or halogen atoms. This has hitherto been explained on the assumption that such substituents sterically hinder the reactions of the carbonyl group in the ortho-position to which they lie. It appeared to the authors that a better explanation might be found in the influence of the substituents on the isorropic process in which the quinone carbonyls are concerned.

An examination was made of the absorption spectra of the following quinones:—*p*-Benzoquinone, toluquinone, *p*-xyloquinone, thymoquinone, chlorobenzoquinone, bromobenzoquinone, 2:6-dichlorobenzoquinone, trichlorobenzoquinone, trichlorotoluquinone, dibromothymoquinone, and dichlorothymoquinone; and from these spectra the following facts may be deduced:—(1) Benzoquinone has an isorropic band of long persistence, and shows no sign of benzenoid structure; (2) the introduction of methyl radicles or of halogen atoms tends to diminish the persistence of the isorropic band, and to produce in the spectrum a benzenoid band, the change being greater with chlorine than with methyl; (3) the benzenoid character of the compound is intensified in proportion to the decrease in the isorropic band.

From these facts the following conclusions may be drawn:—Benzoquinone exists almost entirely in the quinonoid form. Toluquinone, while existing to a great extent in the quinonoid form, possesses certain characteristics of benzene, and probably vibrates in a manner similar to that in which the benzene molecule oscillates. Chlorobenzoquinone, although still possessing certain quinonoid properties, is in a state of vibration approximating more closely to the intra-molecular motions of benzene. Trichlorobenzoquinone, in which the isorropic process is practically non-existent, vibrates in a manner closely resembling the vibrations of the benzene ring.

Now Kehrman has shown that the introduction of methyl radicles or halogen atoms hinders the formation of quinoneoximes, and the amount of hindrance observed by him is in complete accord with the change in character from the quinonoid to the benzenoid type which is proved by the spectroscopic evidence. In their previous papers (*Proc.*, xxii., p. 33), the authors showed that in order to bring about the isorropic process the quinonoid structure had first to be produced. As the reactivity of the carbonyl group depends on the isorropic process, it is evident that the benzenoid character of the compounds examined is alone sufficient to explain their non-reactivity, without introducing the question of a purely hypothetical "steric hindrance." The facts now submitted, in conjunction with those already communicated to the Society, tend to show that there is no necessity for the assumption of any such purely mechanical causes underlying the hindrance to chemical reactions.

61. "The Constitution and Properties of Acyl Thiocyanates." By JOHN HAWTHORNE.

It has been shown (Dixon and Hawthorne, *Trans.*, 1905, lxxvii., 468) that when acetyl "thiocyanate" interacts with aniline, two changes occur simultaneously:—(1) Double decomposition, the sulphur appearing as thiocyanic acid, $\text{AcSCN} + \text{PhNH}_2 = \text{HSCN} + \text{AcNHPh}$; (2) addition, the sulphur now exhibiting thiocarbimide functions, $\text{AcNCS} + \text{PhNH}_2 = \text{AcNH}\cdot\text{CS}\cdot\text{NHPh}$. At low temperatures the former reaction predominates, and at high temperatures the latter.

It has now been shown that, besides temperature, two other factors may have an influence on the course of the interaction between an acyl "thiocyanate" and a base:—

(1) The nature of the base presented, and (2) the nature of the acidic radicle of the "thiocyanate." The results are recorded of experiments made at various temperatures on the systems *o*-toluidine—acetyl "thiocyanate" and aniline—propionyl "thiocyanate," from which it appears (1) that *o*-toluidine causes a great increase of the thiocarbimide functions of acetyl "thiocyanate" as compared with aniline, and (2) that, towards aniline, acetyl and propionyl "thiocyanates" behave very nearly alike.

Since in all the above reactions acetyl "thiocyanate" behaves towards a given base as if it were a mixture of acetyl thiocyanate and acetylthiocarbimide in proportions depending on the temperature, a series of determinations of the molecular refraction of the pure substance was carried out. Within the limits of temperature examined, this value was practically constant, the mean, 45.9, as deduced from the formula $M = \frac{(\mu - 1)m}{d}$, being incon-

sistent with the value required for a compound having the structure $\text{Ac}\cdot\text{S}\cdot\text{C}\cdot\text{N}$, but agreeing closely with that corresponding to $\text{Ac}\cdot\text{N}\cdot\text{C}\cdot\text{S}$.

62. "A Mode of Formation of Aconitic and Citrazinic Acids and their Alkyl Derivatives, with Remarks on the Constitution of Aconitic Acid." By HAROLD ROGERSON and JOCELYN FIELD THORPE.

By the condensation of the sodium derivative of ethyl cyanoacetate with ethyl oxalacetate, ethyl α -cyanoaconitate, $\text{CN}\cdot\text{CH}(\text{CO}_2\text{Et})\cdot\text{C}(\text{CO}_2\text{Et})\cdot\text{CH}\cdot\text{CO}_2\text{Et}$, is produced. This ethyl salt on hydrolysis with acid hydrolysing agents is converted into aconitic acid, whilst with alkaline hydrolytic agents it is transformed into citrazinic acid. Alkyl derivatives of these compounds are formed when alkyl derivatives of ethyl α -cyanoaconitate, prepared either by the direct alkylation of this substance or by the condensation of the sodium derivative of ethyl cyanoacetate with alkyl derivatives of ethyl oxalacetate, are hydrolysed. It was shown that aconitic acid, like glutaric acid, has a symmetrical structure, and that α -methylaconitic and γ -methylaconitic acids are tautomeric.

(To be continued).

THE NATIONAL PHYSICAL LABORATORY.

THE Annual Meeting of the General Board of the National Physical Laboratory took place at Bushy House on Friday, March 16, 1906. There were present, in addition to the Chairman, Lord Rayleigh, the following among others:—Sir John Wolfe Barry, Mr. Beilby, Mr. Kempe, Mr. R. K. Graye, Col. Crompton, Mr. Hadfield, Mr. Gavey, and Mr. Howard.

In opening the proceedings Lord RAYLEIGH referred to the great loss the Laboratory had sustained by the deaths of Sir E. Carbutt and Sir B. Samuelson.

The Report of the Executive Committee for 1905 was presented and approved for presentation to the Royal Society, on the motion of Sir J. WOLFE BARRY, seconded by Mr. DAVID HOWARD. The Scheme of Work for 1906 was also approved. The Report showed progress in all directions.

Some fourteen Scientific papers of importance have been published officially, while members of the staff have contributed nine others to various journals.

The second volume of Collected Papers is in course of preparation.

The Scheme of Work for 1906 includes a research into the resistance of materials of construction to impact, the continuation of the wind pressure, and steam researches, the completion of the work with the Ampère balance, and some experiments of great interest on the effect of the continued application of high pressure to insulators. In the

Metallurgical Division a research into the properties of aluminium-bronze promises interesting results.

The Report announced the intention of the Government, communicated to the Royal Society in December last, to grant a sum of £5000 for buildings during the year, and the increase of the Annual Grant by £500. It referred also to the very successful meeting in the House of Commons last August, under the Chairmanship of Mr. Haldane, which led up to a petition signed by 150 Members of the House asking that the grants should be increased, and the Chairman was able to announce that the Chancellor of the Exchequer had recently intimated his intention of making the building grant for the year £10,000 instead of £5000 as originally contemplated.

It was also stated that the Goldsmiths' Company had very generously made a donation of £1000 with the request that it should be devoted to some specific object.

The very cordial thanks of the Board were voted to the Chancellor of the Exchequer, Mr. Haldane, Sir J. Lawrence, Sir J. Brunner, and the other gentlemen who had interested themselves in the House of Commons petition, and also to the Goldsmiths' Company.

The Director gave an account of the proposed additions to the buildings rendered possible by the increased grant, and explained the plans which had been prepared by the Building Committee. The suggestion that the work of erecting these buildings should now be pressed forward was cordially welcomed, and at a meeting of the Executive Committee held later power was given to the Building Committee to take the necessary steps.

The Board then adjourned to inspect the Laboratory and to view the new Electrical Buildings. These are now approaching completion. They have been erected by Messrs. Mowlem and Co., at a cost of about £8000, to the design of Messrs. Mott and Hay, who very kindly gave their services, while with marked generosity Messrs. Mowlem's tender was based on the cost price of the buildings.

It is hoped that they may be opened on June 25th, on the occasion of the visit of the foreign guests of the Institution of Electrical Engineers. In view of this ceremony the invitations on Friday were restricted to Members of the Board and their personal friends; the usual annual gathering of friends of the Laboratory being postponed until June.

NOTICES OF BOOKS.

Utilisation of Nitrogen in Air by Plants. By THOMAS JAMIESON. Aberdeen: Agricultural Research Station. 1905.

THE author of this book claims that he has made a discovery which must be recognised as of the greatest importance—namely, that plants in general possess the power of absorbing nitrogen from the atmosphere. Cereals and grasses have the same power, though to a less extent than other plants. He asserts that the opposite conclusion was based upon experiments performed on very small and weak plants, grown under such unnatural conditions that full vigour was not attained. He first brings forward arguments to disprove the tubercle theory, and then proceeds to state the reasons which have led him to adopt the conclusion above referred to. He bases his views upon the discovery of specialised structures to which he gives the name "albumen generators." These structures are found on the softest parts of the younger leaves and leaf stalks of many families, among which may be mentioned the weeds *Spergula*, *Stellaria*, and *Urtica*, and many cultivated plants. The argument rests entirely upon the discovery of the presence of albumen (detected by means of iodine, cupric sulphate, and mercury nitrate) in the tip of the hair-like structure and its subsequent discharge into the leaf; and apparently no tests were arranged to demonstrate the

source of the nitrogenous compound. The author suggests practical applications, and describes some trials which have been made to show that the legumes are not alone in benefiting the after crop, rape being suggested as the most suitable plant to grow as a nitrogen provider; but the facts given are quite insufficient to prove the theory.

Electro-chemistry of Organic Compounds. By Dr. WALTHER LÖB. Authorised Translation by H. W. F. LORENZ, A.M., Ph.D. New York: John Wiley and Sons. London: Chapman and Hall, Ltd. 1906.

THE second American edition of Dr. Löb's work has been prepared from the third German edition, which the author published about a year ago, and which was greatly enlarged and thoroughly revised. The new matter, which attains the same high degree of excellence as was a marked feature of the earliest edition, includes some sixty pages on Electro-thermic Processes and the Silent Electric Discharge, in which a brief *résumé* of the subject is given. A new chapter on Electric Endosmose or cataphoresis describes the effects of this phenomenon and touches upon its practical importance and application. Considerable alteration has been made in the arrangement of the text, the general theory of electrolysis being treated in an introductory section, in which the theoretical aspects of organic electro-chemistry are discussed more fully than in the old edition, while some space is devoted to the discussion of methodics, although the author has not aimed at providing a laboratory guide, and merely wishes to indicate some of the more important details of the practical work in such a way as to enable the reader to understand thoroughly the various methods commonly employed.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances de l'Académie des Sciences. Vol. cxlii., No. 9, February 26, 1906.

Difficulties of Estimation of Carbon Monoxide in Gaseous Mixtures.—Armand Gautier and M. Clausman.—The estimation of small traces of carbon monoxide in a mixture of such gases as ethylene, methane, nitrogen, hydrogen, oxygen, &c., is attended with great difficulties. The authors point out a number of these difficulties and various methods of overcoming them.

Addition of Hydrochloric Acid to Isobutylene Oxide, $(\text{H}_3\text{C})_2\text{C}(\text{O})\text{CH}_2$.—Louis Henry.—The author's researches prove beyond doubt that there do exist two distinct isobutylenic compounds, due to the considerable functional difference existing in the carbon atom, owing to the presence or absence of hydrogen. The existence of these two chlorohydrins being established, the author is proceeding to prove that hypochlorous acid, $(\text{HO})\text{Cl}$, can be fixed on to isobutylene, $\text{H}_3\text{C}-\text{CH}=\text{CH}_2$.

Lævo-lactic Acid.—E. Jungfleisch and M. Godchot.—The authors describe the preparation of pure crystalline *l*-lactic acid, and investigate its properties. They find that solutions of *l*-lactic acid when charged with *l*-lactylactic acid can at the same time give solutions of zinc salts of the two acids, zinc *l*-lactylactate having a dextro-rotatory power considerably greater than that of zinc *l*-lactate. These facts, which are analogous to those which have been observed for *d*-lactic acid, may have an influence on the exact determination of the nature of lactic acids resulting from numerous microbe fermentations.

MEETINGS FOR THE WEEK.

- MONDAY, April 2nd.**—Society of Arts, 8. (Cantor Lectures). "Fire Risks, and Fire Extinction," by Prof. Vivian B. Lewes.
— Royal Institution, 5. General Monthly Meeting.
— Society of Chemical Industry, 8. Election of Officers and Committee. "Ropiness in Flour and Bread, and its Detection and Prevention," by E. J. Watkins. "The Röse-Hersfeld and Sulphuric Acid Methods for the Determination of the Higher Alcohols (a Criticism)," by V. H. Veley.
- TUESDAY, 3rd.**—Royal Institution, 5. (Tyndall Lectures). "The Influence of Geology on Scenery," by John E. Marr, F.R.S., &c.
- WEDNESDAY, 4th.**—Society of Arts, 8. "Ramie and its Possibilities," by Mrs. Ernest Hart.
- THURSDAY, 5th.**—Royal Institution, 5. "Internal Combustion Engines" (with Experimental Illustrations), by Prof. Bertram Hopkinson, M.A., &c.
— Chemical, 8.30. "An Improved Apparatus for Measuring Magnetic Rotations and obtaining a Powerful Sodium Light," by W. H. Perkin, sen. "Rusting of Iron," by G. T. Moody. "Determination of Carbon in Soils," by A. D. Hall, N. H. J. Miller, and N. Harmer. "The Electrolysis of the Salts of $\beta\beta$ -Dimethylglutaric Acid," by J. Walker and J. K. Wood. "Bromo- and Hydroxy-derivatives of $\beta\beta\beta\beta$ -Tetramethylsuccinic Acid," by J. K. Wood. "Some New Orthoxylester Derivatives," by G. Stallard. "New Solvent for Gold," by J. Moir. "Molecular Condition in Solution of Ferrous Oxalate" (a Correction), by S. E. Sheppard and C. E. K. Mees.
- FRIDAY, 6th.**—Royal Institution, 9. "The Physical Basis of Life," by William Bate Hardy, F.R.S., &c.
- SATURDAY, 7th.**—Royal Institution, 3. "The Corpuscular Theory of Matter," by Prof. J. J. Thomson, F.R.S., &c.

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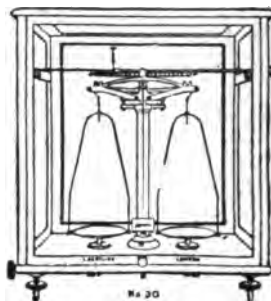
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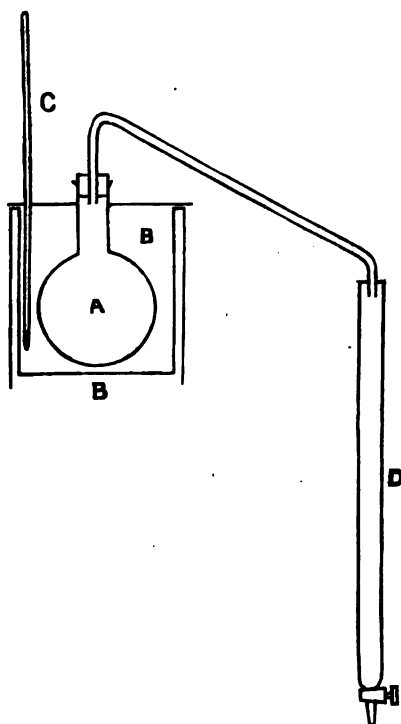
VOL. XCIII., No. 2419.

ROSIN SIZE ANALYSES.

By CLAYTON BEADLE and HENRY P. STEVENS.

ROSIN is the residue left in the retort when crude turpentine is distilled with the object of obtaining "oil" or "spirits of turpentine." It is not to be expected that the separation of the rosin from the spirits of turpentine would be complete, and a trace of the latter remains behind in the rosin—the quantity varying with the form of plant used for distilling and the temperature to which the crude turpentine is heated.

When the rosin is used for making rosin size, it will therefore contain a residue of crude turpentine. In the manufacture of rosin size this turpentine may be partially driven off or the greater part retained, according to the system employed; and the final product, the rosin size, may also contain larger or smaller quantities of turpentine.



DIRECT DETERMINATION OF MOISTURE IN ROSIN SIZE APART FROM OTHER VOLATILE BODIES.

A, Flask heated in asbestos air-bath, B, provided with thermometer, C. Water and other volatile substances collect in the burette D, where the volume is read off.

To estimate the moisture in rosin size, it is usual to dry down a small quantity in a flat dish or clock-glass kept at a constant temperature of 100° to 110° C.; the loss in weight is taken to represent the moisture. Should, however, the size contain a large proportion of turpentine, part or whole of this may be driven off and lost, and will therefore be included as moisture.

With the object of testing this point, a very thorough analysis was made on a sample of rosin size by different methods, and the results compared. We are indebted to

Mr. G. W. Ross for carrying out these experiments, which are recorded below. The close agreement between the results obtained by different methods speaks for the accuracy of the determinations. The moisture was determined by the following methods:—

1. As suggested by Mr. H. Ardleter, attempts were made to estimate the water directly by distillation. The method adopted by us was the following:—A quantity of size was weighed into a glass flask, which was then placed in an air-bath and connected with a condensing tube, the end of which was bent over, dipping an inch or so into a burette graduated in tenths of a c.c. The burette, before the experiment, was filled up with water, which was then allowed to run out until the 50 c.c. mark at the bottom of the tube was reached. The flask was then gradually heated in the air-bath, and the distillate which condensed in the tube collected in the burette. The heating was gradual, and the distillation took half an hour to an hour. When nothing further distilled over, the volume of the distillate was read off. In each case a small quantity of turpentine was found floating on the surface of the water, and the volume of this was read off separately.

2. At the end of the experiment the flask was allowed to cool and then re-weighed, thus giving the loss in weight by difference. This loss would represent moisture, turpentine, and any gaseous substances driven off.

3. The loss by difference was checked by a test carried out in the ordinary method by drying down at 100–110° C. a weighed quantity of the size in a clock-glass.

4. A complete analysis was made of the size, including the total rosin, total soda, other mineral matter, and turpentine. The remainder will represent moisture "by difference." The figures obtained were as follows:—

METHODS 1 and 2.

Experiment.	Volume of water distilling over.	Loss in weight of contents in flask.
	Per cent.	Per cent.
1.	38.5	39.8
2.	39.8	39.4
3.	38.0	39.4
4.	38.7	39.6
Mean of all four experiments ..	38.75	39.55

METHOD 3.

Loss of weight by drying down in a clock-glass at 100–110° C.	39.5
--	------

METHOD 4.—Rosin by Extraction with Ether.

Experiment.	Total.
	Per cent.
1.	55.8
2.	56.3
3.	56.0
Mean of three experiments ..	56.03

Complete Analysis.

Total rosin	56.03
Total soda (Na ₂ O)	3.55
Turpentine	0.34
Other mineral matter	0.24
Moisture (by difference)	39.84
	100.00

It will be seen that the amount of turpentine distilled over is very small—on an average only 0.34 per cent. The figures for moisture obtained by different methods agreed very well among themselves. The figures obtained in Method 1 vary more among themselves than some of the others, as there is a limit of accuracy in the direct estimation of the moisture, as it must be remembered, in the first place, that an ordinary burette cannot be read to less than one-twentieth of a c.c.; and then, again, it is not

practicable to take very large quantities of the size. The amounts actually taken were as follows:—

Experiment 1	37.70	grms. taken for analysis.
" 2	36.27	" " "
" 3	14.61	" " "
" 4	54.76	" " "

There is another practical difficulty in estimating the moisture directly in this manner. The water and turpentine distilling over have to pass through a condensing tube before reaching the burette where they are measured. When a dry condensing tube is taken to start with, a certain amount of moisture remains adhering to the sides. To get this into the burette the tube must be carefully warmed, so as to increase the mobility of the water and allow it to run down into the burette. A small loss of moisture under these circumstances is unavoidable. In two of these experiments another method was adopted. The condensing tube was rinsed out with water before starting the distillation, on the assumption that it would contain at the start of the experiment as much water as it would retain at the end. It is curious to note that the turpentine seems to come over with the first drops of water, although its boiling-point is considerably above that of water.

Turpentine is a mixture of various substances, and its main constituent, "pinene," boils at 155–156° C.; that is, considerably above the boiling-point of water. The quantity of turpentine-like bodies obtained was too small to allow of further examination. The turpentine was estimated by measuring its volume and calculating its weight by allowing for its density (0.86 to 0.87).

The results obtained are, taken as a whole, very concordant. The figure for moisture (39.5), obtained by drying down on a watch-glass in the ordinary method, agrees very well with that obtained in a similar manner by the mean loss in the weight of the flask, viz., 39.55. The other figures are almost as close, 38.75 being the mean percentage for the volume of water distilled over, and 39.84 for moisture by difference on the complete analysis. This latter figure is, perhaps, higher on account of the total rosin being possibly too low; it is always difficult to extract the last portions of resin with ether unless the operation be repeated several times with fresh portions, due to the solubility of ether in water. We may conclude that, so far as the results go, no sensible error is produced by determining the moisture by drying down a small quantity in an open vessel, such as a clock-glass; but this might not hold with samples containing more turpentine than that used for these experiments. This point can only be settled by subjecting a number of samples of rosin size from different sources to distillation and other tests, as above.

Royal Institution.—The following are the Lecture Arrangements at the Royal Institution after Easter:—Professor G. Baldwin Brown, two lectures on "Greek Classical Dress in Life and in Art"; Professor William Stirling, three lectures on "Glands and their Products"; Dr. O. Chalmers Mitchell, two lectures on "The Digestive Tract in Birds and Mammals"; Rev. J. P. Mahaffy, two lectures on—(1) "The Expansion of Old Greek Literature by Recent Discoveries," (2) "The Influence of Ptolemaic Egypt on Græco-Roman Civilisation"; Professor William J. Sollas, three lectures on "Man and the Glacial Period"; Professor Charles Waldstein, three lectures on "English Furniture in the Eighteenth Century"; Professor Sir James Dewar, two lectures on "The Old and the New Chemistry"; and Professor W. Macneile Dixon, two lectures on—(1) "The Origin of Poetry," (2) "Inspiration in Poetry." The Friday Evening Meetings will be resumed on April 27, when Professor John W. Gregory will deliver a Discourse on "Ore Deposits and their Distribution in Depth." Succeding Discourses will probably be given by the Hon. Charles A. Parsons, Professor J. H. Poynting, Professor Arthur Schuster, Mr. Leonard Hill, Professor H. Moissan, Professor Sir James Dewar, and other gentlemen.

A SYSTEM OF QUALITATIVE ANALYSIS INCLUDING NEARLY ALL THE METALLIC ELEMENTS.*

PART I. (continued).—PREPARATION OF THE SOLUTION
(INCLUDING THE DETECTION OF OSMIUM, SILVER,
AND LEAD).

By ARTHUR A. NOYES.

(Continued from p. 149).

Procedure 5.—Loosen the dehydrated residue (P. 2, 3, or 4) from the dish, and rub it to a fine powder with a pestle; add to it 5 c.c. HNO_3 (1:20), or 10 c.c. if it seems necessary, cover the dish, and heat on a steam-bath for ten minutes. Note whether there is any residue whatever. If no residue can be seen, rub the sides of the dish with the rubber-covered end of a glass rod, and allow the liquid to stand three or four minutes. If there is a crystalline residue of nitrates, add enough water to dissolve it, and note whether an amorphous residue remains. Dilute with 20 c.c. water, and heat to boiling. If the substance was a non-metallic one, unite at this point the two portions (treated by P. 2 and P. 3), unless it is desired to know their constituents separately. Decant the solution through a washed filter, retaining in the dish as much of the residue as possible. If the residue is large in amount, heat it again with 5 c.c. HNO_3 (1:20), dilute with water, and decant the solution through the same filter. Wash the residue with HNO_3 (1:05), decanting the washings through the filter, and transfer it to a platinum dish. (Residue, P. 6; solution, P. 51).

Notes.

1. If SiO_2 has not been added, important conclusions can be drawn in regard to the absence of certain elements in the substance when nothing whatever remains undissolved upon treating the dehydrated residue with HNO_3 (1:20). The fact must not be overlooked, however, that in the dehydrated form even a very small residue or slight turbidity may correspond to an appreciable quantity of an element. A perfectly limpid solution and one that does not deposit any sediment whatever after a few minutes' standing must be obtained, if such conclusions are to be drawn. Moreover, it should be noted whether such a solution results before diluting the HNO_3 (1:20) with water (or after diluting it with as little water as suffices to dissolve crystalline nitrates); for any residue is more difficult to recognise in a large volume of liquid.

In case no residue whatever remains, it shows the absence of silicon, tin, niobium, and tantalum in quantity as large as 1 m.grm.; of antimony in quantity as large as 2 m.grms., except when an organic hydroxy-acid is present, and of tungsten in quantity as large as 1 m.grm., except when phosphoric or arsenic acid or an organic hydroxy-acid is present. It also indicates the probable absence of titanium in quantity as large as 1 m.grm., except when zirconium is present.

Organic hydroxy-acids, like tartaric, lactic, and citric acids, dissolve Sb_2O_3 and H_2WO_4 with great readiness. H_2WO_4 also dissolves very readily when H_3PO_4 or H_3AsO_4 is present, owing to the formation of complex acids. The presence of arsenic, phosphoric, and organic acids is detected in the course of the subsequent procedure. Boric acid does not increase the solubility of any of the oxides of the tungsten and niobium groups. Moreover, it will not be present if the substance has been treated with HF; for even 1 m.grm. H_3BO_3 volatilises completely upon evaporation with 5 c.c. of this acid and 5 c.c. HNO_3 (1:42). Even 500 m.grms. of titanium may dissolve completely in the HNO_3 when much zirconium (500 m.grms.) is present.

2. If SiO_2 has been added, the presence or absence of the tungsten and niobium groups cannot be determined

* From the *Technology Quarterly*, xvi., No. 2.

until the residue insoluble in HNO_3 has been treated with HF , the solution evaporated with H_2SO_4 and NH_4OH and $(\text{NH}_4)_2\text{S}$ added (P. 6 and Part II.).

3. The residue insoluble in HNO_3 may contain, in addition to the oxides of the elements named in N. 1, the following substances:— MoO_3 , when molybdenum is present in quantities over 100 m.grms., or when tungsten or tin is also present; GeO_2 , when over 30 m.grms. Ge are present; Bi_2O_3 , when much antimony is also present; SeO_2 and TeO_2 , when much tin or antimony is also present; ThF_4 , when much thorium is present in the residue treated with HF ; the phosphates and arsenates of tin, titanium, zirconium, and thorium, and the vanadate of tin, when the components of these substances are simultaneously present. The residue may also contain certain undecomposed substances (named in P. 6, N. 2) which are not attacked by either HNO_3 or HF .

4. Of the elements named in N. 3 which may under the specified conditions be present in the residue insoluble in HNO_3 , the following ones always pass into the HNO_3 solution in considerable proportion when large quantities of them are present:—Molybdenum, bismuth, selenium, tellurium, zirconium, thorium, vanadium, germanium. This is also true of titanium (N. 1). Furthermore, a small quantity of antimony (1–2 m.grms.) dissolves. Phosphoric, arsenic, and vanadic acids are dissolved completely when tin, titanium, and zirconium are absent.

5. Aside from the elements named in N. 1, and under the specified conditions aside from those named in N. 3, no others are retained in the residue insoluble in HNO_3 in important quantity, as far as is known. Even the elements bismuth, tellurium, zirconium, and thorium, whose salts are readily hydrolysed, dissolve completely when present alone in quantities up to 500 m.grms. This is also true of zirconium, even when 500 m.grms. Ti are present. The elements iron, aluminium, and chromium, whose oxides are made insoluble by ignition, dissolve completely when the residue has been heated only to 120° , at any rate upon the second digestion with HNO_3 (1:20). Of the element gold, if originally present as chloride, only the small portion reduced by dust from the air is left in the residue.

6. Whenever it is directed, as at the end of this procedure, to "wash" a residue or precipitate, it is understood, unless otherwise stated, that this operation is to be continued until the washings will give no test for some substance known to be present in the solution in considerable quantity.

Procedure 6.—Support the filter containing some of the residue insoluble in HNO_3 (P. 5) in a stout platinum ring or in a funnel completely covered with paraffin. Pour through it 5–10 c.c. pure concentrated HF , collecting the liquid in the platinum dish containing the main part of the residue. Heat on a steam-bath for five minutes. Dilute with 25 c.c. cold water, decant the solution through the same filter, and wash the residue and the filter thoroughly. Then rinse the residue from the platinum dish into a casserole. Collect the solution in a second platinum dish. (Residue, P. 7; solution, P. 31).

Notes.

1. The HF dissolves silica and all the oxides of the niobium and tungsten groups, except that of tin, when this is present in large quantity; but this also dissolves on diluting the solution with cold water. All the substances specifically named in P. 5, N. 3 also pass into solution in HF , with the exceptions of ThF_4 and $\text{Th}_3(\text{PO}_4)_4$, of the Bi_2O_3 present in excess of about 6 m.grms., and of some $\text{Sn}_3(\text{PO}_4)_4$ when this is present in large quantity.

2. A residue undissolved by the HF may consist of one or more of the following substances, if the original substance was non-metallic:— PbSO_4 , BaSO_4 , SrSO_4 ; HgS , native MoS_2 ; MnO_2 , PbO_2 ; ignited or native Al_2O_3 , Cr_2O_3 , FeCr_2O_4 (chromite), SnO_2 , or TiO_2 ; AgCl , BiF_3 , ThF_4 , Prussian blue, $\text{Sn}_3(\text{PO}_4)_4$, $\text{Th}_3(\text{PO}_4)_4$

(as in monazite), certain silicates (such as cyanite, andalusite, tourmaline, beryl, zircon, topaz), S, C, SiC. It may consist of one of the following substances, if the original substance was a metal:—Au, Pt, Ir, Rh, Ru, or Os, or alloys of these, or certain alloys of iron (such as ferrochrome, ferrosilicon).

3. In spite of the rather numerous exceptions named in the preceding note, by far the larger proportion of substances ordinarily submitted to analysis, including almost all silicates, are decomposed by treatment according to the foregoing procedures.

Procedure 7.—Add to the residue insoluble in HF (P. 6) 15 c.c. HCl (1:20), and boil gently for five minutes. Then add slowly 10 c.c. water, keeping the liquid at its boiling-point. Decant while still hot through the filter previously used, if this contains any of the residue. [If it is doubtful whether the HCl has dissolved anything, evaporate the solution to dryness to determine it]. If the residue has been dissolved to a considerable extent, but not completely, boil it with successive portions of HCl (1:20) as long as a considerable quantity of substance is dissolved, diluting and decanting each time as before. To the residue add 3 c.c. HNO_3 (1:42), and 10 c.c. HCl (1:20), and heat on a water-bath for five minutes, or longer if an action is taking place. Dilute with an equal volume of water, filter through the same filter-paper, and wash the residue. Unite the HCl and aqua regia solutions. Separate the residue from the filter, or incinerate the latter if necessary. (Residue, P. 8; solution, P. 17).

Notes.

1. Of the substances named in P. 6, N. 2, which may constitute the undecomposed residue, it will be found that PbSO_4 , SrSO_4 , MnO_2 , and PbO_2 dissolve completely in 30 c.c. HCl (1:20) on five minutes' boiling, even when such quantity is present as contains 500 m.grms. of the metallic element, and they remain in solution on the addition of 20 c.c. boiling water. The same is true of fused AgCl up to a quantity containing 100 m.grms. Ag, of BaSO_4 up to 20 m.grms., and of $\text{Sn}_3(\text{PO}_4)_4$ in large quantity.

2. By the treatment with aqua regia, HgS , Au, and Pt (except when alloyed with another platinum metal) are also completely dissolved.

3. The cases in which a subsequent fusion must be resorted to are therefore rare. The important substances for which it is necessary are named in P. 8, N. 1 and 2.

Procedure 8.—If the residue insoluble in aqua regia (P. 7) is non-metallic and light coloured, and if it is thought to consist of a silicate or of BaSO_4 , or if there is no indication as to its nature, fuse it with Na_2CO_3 as directed in P. 9.

If the residue is non-metallic and light coloured, and if it is thought to consist of a metallic oxide, fluoride, or phosphate, fuse it with $\text{K}_2\text{S}_2\text{O}_7$ as directed in P. 12.

If the residue is metallic, or if it is non-metallic and dark coloured, fuse it with Na_2O_2 as described in P. 13.

Notes.

1. The important light coloured non-metallic substances which may occur in the residue insoluble in HF , HCl , and aqua regia are:— BaSO_4 , certain silicates, ThF_4 , native or ignited Al_2O_3 , TiO_2 , $\text{ZrO}_2\cdot\text{SiO}_2$, monazite, and SnO_2 . By fusion with Na_2CO_3 the silicates and BaSO_4 are readily converted into soluble compounds, but this method is ineffective with the other substances (except possibly ThF_4). All of these, except SnO_2 , dissolve in dilute H_2SO_4 after fusion with $\text{K}_2\text{S}_2\text{O}_7$. SnO_2 is best brought into solution by fusing it with twenty times its weight of a mixture of equal quantities of Na_2CO_3 and S in a porcelain crucible, extracting the mass with water, and treating the solution according to Part II., and the residue, after dissolving in hot HCl (1:12), and properly diluting by P. 71.

2. The important substances that may constitute a

dark coloured non-metallic residue are :—Chromite (FeCr_2O_4), graphite or coal (C), carborundum (SiC), molybdenite (MoS_2), and Prussian blue ($\text{Fe}_4(\text{FeC}_6\text{N}_6)_3$). A metallic residue may consist of an alloy of iron (like ferrosilicon or ferrochrome), or of a platinum metal or alloy. Most of these substances require fusion with an oxidising flux, and all of them yield to it. The extremely powerful one, Na_2O_2 , is employed so that the method may be general; for even the platinum metals, if in a moderately fine state of division, are oxidised by it. A mixture of Na_2CO_3 and KNO_3 would effect decomposition of the other substances, and, if preferred, it may be substituted for Na_2O_2 , except when platinum metals are to be treated.

Procedure 9.—If its character makes fusion with Na_2CO_3 advisable (P. 8), mix the residue insoluble in aqua regia (P. 7) with ten times its weight of anhydrous Na_2CO_3 in a platinum crucible. Cover the crucible, and heat it for thirty minutes either over a powerful burner (preferably within a cylindrical porcelain jacket) or over a blast-lamp, adding more Na_2CO_3 if a perfectly fluid fusion does not result. Insert in the liquid mass a stout platinum-wire with a small horizontal ring at the lower end, and allow the mass to solidify completely. Then heat the crucible till the mass begins to melt around the sides, and remove it by means of the platinum-wire. Digest it on a steam-bath with 50 c.c. water till the mass is entirely disintegrated. Filter, and wash the residue. (Residue, P. 10; solution P. 11).

Notes.

1. If the mass is not removed from the crucible, its disintegration by the hot water is often very slow.

Procedure 10.—Heat the residue from the aqueous solution of the fused mass (P. 9) with 5 c.c. HCl (1:12) for five minutes, and add 25 c.c. hot water. Filter, and wash the residue. (Residue, P. 12; solution P. 71).

Notes.

1. Of the elements of the tungsten, niobium, selenium, and silver groups, tin and titanium are the only ones that are at all likely to be present in the HCl solution of the fusion of the residue (P. 7) insoluble in all the acids. As these elements are tested for also in connection with subsequent groups the solution is immediately precipitated with H_2S (P. 71).

Procedure 11.—Acidify the aqueous solution of the fused mass (P. 9) with HCl , evaporate the solution to dryness, and heat the residue in a hot closet at 120° for an hour. Heat the residue with 5 c.c. HCl (1:12). Add 25 c.c. water, filter, and wash the residue. (Residue, P. 6; solution P. 71).

Notes.

1. The solution of the fused mass is evaporated with HCl , and the residue heated at 120° and treated with HCl to separate silica, which is very likely to be present. A white insoluble residue probably consists of nothing but silica, but, if desired, this may be determined by warming it with HF (P. 6), and treating the solution by the subsequent procedures (P. 31).

2. Since it is very improbable that elements of the selenium and silver groups are present, owing to the fact that most, if not all, of their compounds are decomposed by the preceding treatment with various acids, the solution of the dehydrated residue is precipitated at once with H_2S .

Procedure 12.—If the character of the residue insoluble in aqua regia (P. 7) makes fusion with $\text{K}_2\text{S}_2\text{O}_7$ advisable (P. 8), or if this residue has been fused with Na_2CO_3 and undecomposed substance still remains on treating the fused mass with HCl (P. 10), pour the residue gradually into a platinum crucible containing ten times as much $\text{K}_2\text{S}_2\text{O}_7$ in a state of fusion. Cover the crucible, and keep the temperature at a dull red heat for twenty minutes. Cool the crucible, pour into it 2–4 c.c. H_2SO_4 (1:84) and heat

again, keeping the crucible covered until the whole mass becomes fluid. Cool the crucible, and gradually pour its (still liquid) contents into 30 c.c. water. Decant the solution from the residue, add to this 5–10 c.c. H_2SO_4 (1:20), and heat to boiling. Filter, and wash the residue. (Residue, P. 6, and if there is still a residue, treat it by P. 20). Unite the filtrate with the previously decanted solution. Add 2 drops HCl (1:12), shake the solution vigorously for five minutes with 1 c.c. moist precipitated Ag , and filter. (Precipitate, reject; filtrate, P. 71).

Notes.

1. The 2–4 c.c. H_2SO_4 are added at the end of the fusion to save the considerable time otherwise consumed in the disintegration of the mass by water, to dissolve as far as possible rare earth phosphates (as in monazite) which otherwise may separate in large quantity on the addition of water, and to hold titanium in solution, which it does even though the solution be boiled. Any undissolved residue is heated with H_2SO_4 (1:20) in order to dissolve these phosphates and also any double sulphates of the rare earths (like $2\text{K}_2\text{SO}_4 \cdot \text{Th}(\text{SO}_4)_2$) which may have been precipitated in crystalline form.

2. An insoluble residue may consist of SiO_2 , which is ascertained by treating it with HF (P. 6) and evaporating the solution to complete dryness after the addition of a little H_2SO_4 ; of alkaline earth sulphates, which are treated by P. 20, and of undecomposed substances (such as certain silicates, cassiterite, &c.), which would be left as a residue upon boiling with Na_2CO_3 (P. 20), and which should be fused with Na_2CO_3 (P. 9), or with a mixture of Na_2CO_3 and S (P. 8, N. 1).

3. The solution is first shaken with Ag to remove the platinum which is dissolved from the crucible by the acid flux; if this is not removed a precipitate will be obtained with H_2S even though no elements of that group are present. The Ag precipitate is rejected, since it cannot contain mercury (owing to its volatility at a red heat) or gold (owing to the metal being unattacked by $\text{K}_2\text{S}_2\text{O}_7$), and is very unlikely to contain palladium or platinum (aside from that taken up from the crucible), and since these are the only elements precipitable by silver from a solution containing little HCl (see Part IV.).

Procedure 13.—If its character makes fusion with Na_2O_2 advisable (P. 8), introduce the residue insoluble in aqua regia (P. 7), gradually if a violent action occurs, into a nickel crucible in which 5 grms. Na_2O_2 have been brought to a state of fusion. Heat the lower part of the crucible to dull redness, maintaining a temperature sufficient to keep the mass fluid for from five to twenty minutes, according to the readiness with which the residue is attacked. After cooling, place the crucible on its side in a covered casserole, add 50 c.c. cold water, and, after the violent effervescence has ceased, warm gently. Rinse out the crucible, transfer the solution and residue to a flask, and add gradually HCl (1:20) until the black residue of $\text{Ni}_3\text{O}_4 \cdot 2\text{H}_2\text{O}$ dissolves, meanwhile keeping the solution cold. If complete solution does not result, decant the solution, after settling, from the residue, boil the latter with HCl (1:20), and unite the solution and any residue with the decanted solution. Transfer the mixture to a 200 c.c. distilling flask, having its side arm bent downwards and dipping into 10 c.c. 5 per cent NaOH solution in an Erlenmeyer flask surrounded by a large beaker of cold water. Close the flask with a cork through which passes a straight tube reaching to the bottom. Boil the solution for five to ten minutes, taking care that all the distillate condenses. Replace the NaOH solution containing the distillate by a fresh portion of NaOH solution, and distil again for five minutes. (Distillate, P. 14; residual solution, P. 15).

Notes.

1. Of the substances possibly present in a dark coloured or metallic residue, the platinum metals and their alloys are most difficultly attacked by Na_2O_2 . They should therefore be rather finely powdered, which is usually best

accomplished by pounding the substance in a diamond steel mortar. It is not necessary, however, to resort to the special methods of producing an extremely fine state of division, like fusion with lead, which have to be employed when other methods of attack, like heating with NaCl and Cl₂, are to be used. Twenty minutes' fusion is sufficient with 500 m.grms. of the most resistant alloys.

2. As the nickel crucible is much attacked by the flux, the fusion should not be unduly prolonged. Silver and platinum crucibles are also strongly attacked, and have no advantage in this respect, while they have the disadvantage of being much more expensive.

3. The flux Na₂O₂ can now be obtained commercially in a fair degree of purity, being made from metallic sodium; it often contains some iron, however. Nevertheless, to determine the nature and approximate quantity of the impurities introduced from the crucible and flux into the solution to be analysed, a blank fusion and an analysis of the fused mass should be made.

4. Upon treatment of the fused mass with water, a violent evolution of oxygen occurs, and a large quantity of a fine black precipitate consisting of Ni₃O₄·2H₂O separates. This dissolves completely on the addition of HCl.

5. The products resulting from the fusion of the substances (named in P. 8, N. 2) possibly present in the residue, and treatment of the fused mass with water, are as follows:—Chromite, Fe₂O₃ and Na₂CrO₄; carbon, Na₂CO₃; carborundum, Na₂CO₃ and Na₂SiO₃; molybdenite, Na₂MoO₄ and Na₂SO₄; Prussian blue, Fe₂O₃ and Na₂CO₃; ferrosilicon or ferrochrome, Fe₂O₃ and Na₂SiO₃, or Na₂CrO₄; platinum, xNa₂O·Pt₂O₃; iridium, xNa₂O·IrO₃; rhodium, RhO_x(?); osmium, Na₂OsO₅(?); ruthenium, Na₂RuO₅(?). All of these products, except Fe₂O₃, xNa₂O·Pt₂O₃, xNa₂O·IrO₃, and RhO_x, dissolve upon treating the fused mass with water. Na₂CrO₄ imparts to the solution a pure yellow colour, Na₂OsO₅ a yellow-orange colour, and Na₂RuO₅ a dark orange-red colour. All of the products insoluble in water dissolve on the addition of HCl to the solution or on heating the residue with HCl (1:20), except a part of the Pt₂O₃·2H₂O when present in large quantity. The Pt₂O₃·2H₂O produced by action of the acid on xNa₂O·Pt₂O₃ forms a dense yellow powder, which is dissolved only slowly by hot HCl (1:20), or even aqua regia. xNa₂O·IrO₃ dissolves in HCl with production of a dark blue solution.

6. The solution must be kept cold during the neutralisation, since otherwise osmium may be lost in the form of the very volatile OsO₄, which is formed when a Na₂OsO₅ solution is acidified. The solution must not be filtered through paper, as this immediately reduces Na₂RuO₅ with formation of a black deposit.

7. The OsO₄ (osmium tetroxide) set free when the Na₂O₂ solution is acidified distils over with great readiness (usually within two or three minutes), since in the pure state it boils not far from 100°, and since it dissolves in water apparently without change. The vapour has an odour somewhat similar to that of chlorine, and is extremely irritating to the eyes and nose. The distillate assumes a yellow colour when a little osmium, a dark orange colour when much osmium is present.

8. Although a similar volatile oxide of ruthenium (RuO₄) exists, it is not formed when the Na₂O₂ solution is heated with HCl; on the contrary, the Na₂RuO₅ present is reduced with formation of RuCl₃. Consequently no ruthenium distils over.

9. If osmium were present in the original substance in the form of one of its salts, it would be completely volatilised in the first treatment with HNO₃ (P. 1) or in the subsequent evaporation (P. 2 or 3); for all soluble osmium compounds yield OsO₄ when boiled with HNO₃. Therefore, if its presence in such a form be thought possible, the first treatment of the substance with HNO₃ (1:20) should be carried out, as indicated in P. 1, in a distilling flask, the liquid boiled for two or three

minutes, and the distillate collected as in this procedure, and tested by P. 14. As osmium is in practice very rarely met with except in the metallic form, it is, as a general rule, unnecessary to complicate in this way the procedure for the preparation of the solution.

10. Since metallic osmium, both in the pure state and alloyed with iridium or other platinum metal, is scarcely attacked by aqua regia, unless it has been brought to an extremely fine state of division by a chemical method, there is little danger of completely dissolving and volatilising it in the treatment with that solvent in P. 7. Still, if it is desired to determine with certainty the presence or absence of a small quantity of osmium, it is best either to heat the residue undissolved by HNO₃, HF, and HCl with the aqua regia in a distilling flask, and to collect and test the distillate as in P. 13 and 14, or to fuse the residue with Na₂O₂ (as in P. 13) without treating it with aqua regia at all.

Procedure 14.—Add to the distillate (P. 13) four or five drops 20 per cent Na₂S₂O₃ solution, acidify with HCl (1:12), keeping the liquid cool, and add to the solution one-fifth its volume of HCl (1:12). (Precipitate and solution, reject).

Notes.

1. OsS₂ separates as a brownish black precipitate. The test is obtained when only 0.5 m.grm. Os is present in the solution submitted to distillation. It is the volatility of the osmium and its presence in the distillate that is especially characteristic—not the fact of its precipitating with Na₂S₂O₃; for if they could be present in the distillate, other elements—for example, ruthenium—would give a similar precipitate.

Procedure 15.—Evaporate the residual solution from the distillation (P. 13) to dryness, and heat the dry mass at 120° for an hour. Then heat it to boiling with 20 c.c. HCl (1:02). Filter, and wash the undissolved residue. (Residue, P. 16; solution, P. 71).

Notes.

1. The solution is evaporated and the salt mass heated at 120°, so as to make insoluble the H₂SiO₃ and H₂WO₄ that may be present. PtO₃ may also be present in the residue undissolved by the HCl (1:02); see P. 13, N. 5.

Procedure 16.—Treat the residue (P. 15) just as described in P. 6. (Solution in HF, P. 31). Add any residue undissolved by HF to the HCl solution (P. 15), and treat the mixture by P. 71, allowing the cold solution saturated with H₂S to stand an hour or more before filtering.

Notes.

1. Of the elements of the tungsten and niobium groups, only tungsten and titanium are at all likely to be present in this HF solution; for tin and antimony, even if present in the fused residue, would dissolve on treating the dehydrated residue with HCl (P. 15), and niobium and tantalum are not met with at all in alloys nor in compounds insoluble in HF. Hence, the procedure given for those groups may be shortened in its application to this HF solution, by the omission of those operations which serve only to remove and detect the elements almost certainly absent; or this HF solution may be united with the first HF solution obtained when the original dehydrated residue was treated with HF (P. 6).

2. The residue insoluble in HF, if of a yellow colour, may consist of Pt₂O₃. It is added to the solution about to be precipitated with H₂S, because the reagent converts the Pt₂O₃ into Pt₂S₃, which dissolves when the H₂S precipitate is subsequently treated with aqua regia.

Procedure 17.—Evaporate the mixed HCl and aqua regia solutions of the residue insoluble in HNO₃ and HF (P. 7) just to dryness. Heat the residue to boiling with 10 c.c. HCl (1:02), cool, and after two to three minutes filter. Wash the undissolved residue thoroughly with as little HCl (1:02)

as possible, adding the washings to the filtrate. (Precipitate, P. 18; filtrate, P. 71).

Note.

1. The residue undissolved by the HCl (1.02) may consist of AgCl, PbCl₂, PbSO₄, BaSO₄, and SrSO₄, which, though soluble in hot concentrated HCl, are only slightly dissolved by a small quantity of cold HCl (1.02). The solution may contain the other substances named in P. 7, N. 1 and 2, especially lead, manganese, mercury, gold, and the platinum metals. It is therefore united with the main solution and submitted to the regular scheme of analysis, beginning with the H₂S precipitation (P. 71).

Procedure 18.—Pour repeatedly through the filter containing the residue insoluble in dilute HCl (P. 17) a 10—15 c.c. portion of cold NH₄OH (0.90). Filter and wash the residue with NH₄OH (0.96). (Residue, P. 19). Acidify the filtrate with HNO₃. (Precipitate and solution, reject).

Note.

1. The residue is treated with NH₄OH to extract any AgCl present. Cold NH₄OH is used, since PbSO₄ is somewhat soluble in hot NH₄OH. The filtrate is acidified to determine whether any silver is present. The AgCl dissolves in the NH₄OH, owing to the formation of a salt with a complex cation, Ag(NH₃)₂Cl'.

Procedure 19.—Pour repeatedly through the filter containing the residue undissolved by the NH₄OH (P. 18) a 10—20 c.c. portion of hot 25 per cent NH₄C₂H₃O₂ solution to which 5 per cent NH₄OH have been added. Filter and wash the residue with the NH₄C₂H₃O₂ solution until it is free from lead. (Residue, P. 20). Acidify the filtrate with HC₂H₃O₂, and add a few drops of a 10 per cent K₂CrO₄ solution. (Precipitate and solution, reject).

Note.

1. The residue is treated with NH₄C₂H₃O₂ to extract any PbSO₄ or PbO₂H₂ present. Even 500 m.grms. Pb as PbSO₄ dissolve in 15 c.c. boiling NH₄C₂H₃O₂ solution. BaSO₄ is not appreciably soluble. A small quantity of SrSO₄ dissolves, but this does not precipitate with K₂CrO₄ in the confirmatory test for lead. Even if all the SrSO₄ present in the residue passes into the NH₄OH solution there is no danger of failing to detect strontium as a constituent of the original substance; for SrSO₄ is so soluble in dilute HNO₃ (P. 5) and dilute HCl (P. 17) that a considerable quantity of it will be found in the main solution; thus, 10 c.c. of cold HCl (1.04) or of cold HNO₃ (1.032) saturated with SrSO₄ contain about 10 m.grms. Sr.

Procedure 20.—Unite the residue insoluble in NH₄C₂H₃O₂ solution (P. 19) with the residue from the K₂S₂O₇ fusion (P. 12) after its treatment with HF. Transfer these, with the filters if necessary, to a casserole, add 10 c.c. saturated Na₂CO₃ solution, and boil gently for ten minutes, replacing the water which evaporates. Dilute with an equal volume of water, and filter. (Filtrate, reject). Wash the residue thoroughly, and pour repeatedly through the filter containing it a 10 c.c. portion of hot HCl (1.02). (Solution, P. 71).

Notes.

1. Even 500 m.grms. Sr as SrSO₄ are completely converted to SrCO₃ by this procedure. An equal quantity of BaSO₄ would require boiling with two or three successive portions of Na₂CO₃ solution, but as the quantity of BaSO₄ that can be present in this residue cannot be very large, owing to its rather small solubility in HCl (1.20) (see P. 7, N. 1), a single boiling suffices.

2. The Mass Action Law requires that the difficultly soluble sulphate be transformed into carbonate until the ratio (C_{SO₄}/C_{CO₃}) of the concentrations of the SO₄ and CO₃ ions in the solution becomes equal to the ratio (SP_{MSO₄}/SP_{MCO₃}) of the solubility products of the sul-

phate and carbonate in pure water. Now since the second ratio, according to recent solubility determinations, has (at 18°) a value larger than 60 in the case of the two strontium salts, a very large amount of SrSO₄ would have to be transformed to Na₂SO₄ before the ratio C_{SO₄}/C_{CO₃} attained this value. On the other hand, in the case of the two barium salts, the second ratio has apparently the value 0.25 (since it is found that the conversion ceases when C_{SO₄}/C_{CO₃} = 0.25), so that after a certain amount of BaSO₄ has been transformed, the reaction ceases. Aside from this great difference in the equilibrium ratios, the rate of transformation is doubtless much less in the case of the BaSO₄, owing to its very slight solubility. It is to increase this that the solution is boiled.

(To be continued).

THE USEFUL PROPERTIES OF CLAYS.*

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INTRODUCTION.

FROM the earliest dawn of history man has exercised himself in fashioning into products of beauty and utility the plastic materials which he found prepared for his hand on or under the surface of the earth. The stone age was followed by the bronze, and this in time by that of iron, and this in turn by the age of steam and electricity; but throughout all these changes the plastic arts have flourished in proportion to the height of civilisation that the peoples have reached. Science and art, poetry and history combine to lend fascination to a study of clays and clay working. Records of the manners and customs of old and almost forgotten civilisations, such as the Mycenaean, or the Mexican, are carefully gleaned by the archaeologist from the more or less crude linear markings and figures depicted on fragments of pottery and statuary which have come down to us. From the few rude earthenware chips which remain to tell us of our prehistoric ancestors to the finished and delicate porcelains of Dresden and Sèvres, the industry of man records its own development.

Although in the direction of pure utility, as in the manufacture of bricks, tiles, and common earthenware, much has been accomplished, the fact is before us that, in the production of works of art and beauty fashioned from clay, America is not to-day the equal of Europe. The reasons for this are various and need not be discussed here. It is sufficient to point out that they are not of such a nature as to prevent America from ultimately achieving the highest position of all in this domain. The fact that many tons of clay of various sorts are annually brought across the ocean to us for use in our potteries, might seem to indicate that our native clays were of themselves unfit for use. If indeed this were true, it would be an insuperable obstacle to final success, but the fact is that, although it may at present be more profitable to import a clay than to develop a native source, America undoubtedly possesses unusually rich clay and kaolin deposits, some of which are fitted for the production of the most delicate and beautiful objects. It is interesting to note that the pottery which has been a pioneer in works of art made entirely of native clays was founded and fostered by an American woman. It is by just such efforts that wider and further growth will come. In these days of ardent emulation and competition among the nations, it is fair to anticipate the time when an American name shall be added to those which stand for the highest achievement in ceramics, or the art of clay working. As soon as a greater number of individuals among our people become interested in the properties and possibilities of our native clay bodies, development is sure

* United States Department of Agriculture, Bureau of Chemistry, Circular No. 17.

to follow. Clay working is neither so difficult nor so expensive but that in some cases objects of art have been moulded in the home and burnt in private kilns. On the other hand, success is attained only at the price of diligent study and persistent effort. It is with the hope that the dissemination of knowledge concerning the useful properties of clay will lead to a wider popular interest in their study and use, that this article is written.

FORMATION OF CLAYS.

Clay may be defined as an earthy deposit, found on or below the surface, which when finely ground and mixed with water forms a mouldable or plastic mass and can be burned to a hard, stonelike substance after drying. The original crust of the earth was of course formed of the rocks which crystallised from a molten magma. These crystalline rocks of igneous origin underwent gradual crumbling and decay caused by the varied actions of the elements. It is probable that only students of rock structure realise how steadily and continually rocks are undergoing secondary changes and decay. "Enduring as the rocks" is an expression of merely relative truth. When rocks crumble the resulting material may be found either heaped up in places, in which case they are known as residual deposits, or the fine material may have been picked up in suspension by moving waters, sifted out and deposited at some remote place in beds or layers forming sedimentary deposits. These deposits once formed have been, in some cases, acted on by tremendous forces in the course of the great geologic periods. Folded and buried under great crumplings of the earth's crust they have been to varying degrees subjected to heat and pressure and have thus become metamorphosed. Consolidated into sedimentary rocks and shales they become again subject to decay. Changed and disintegrated, now acted on by percolating waters, in which it is known that many of these crystalline minerals are distinctly soluble, or again subjected to acid or alkaline solutions, as the case may be, it is little wonder that every useful earthy deposit found by man on the surface of the earth is to a certain degree unique in its properties, requiring special study and treatment in order to insure the highest degree of success in its working. Clays of nearly the same chemical composition and of similar physical texture, owing to some slight modification will yield entirely dissimilar results in mixing, moulding, and firing. Given in some particular locality a deposit which under certain treatment yields a definite and constant result to man's handicraft, it is doubtful whether, if the deposit becomes exhausted, exactly similar results could be produced elsewhere even by the selfsame artisans. It is to some extent owing to these reasons that the great potteries of the world maintain their distinctive styles, and also lose them as in the cases where the finest modern examples are inferior to the older work. The loss of the art of the ancients, renowned for their pottery and glazes, is possibly due to this cause more than to the inability of modern science and industry to duplicate their cunning in artifice. For the reasons outlined we find our clays with all shades of variation in their useful properties.

PRODUCTION AND IMPORTATION OF CLAYS.

The following figures taken from the statistics of the mineral resources of the United States, compiled by the U.S. Geological Survey for the year 1902, show in an interesting way the condition of our clay-producing industry:—Total imports of clays, 168,551 long tons, valuation 1,154,805 dols.; total domestic production of clays, 1,299,426 long tons, valuation 2,061,072 dols.

Altogether the total amount of clays imported into this country is between one-seventh and one-eighth of that produced here, while the valuation of the latter is less than twice that of the former. Doubtless in time the rich clay deposits of America will be developed to a point where this considerable importation of expensive clays will cease, and one of the factors in this progress will be a better understanding of the useful qualities of clays.

KINDS OF CLAY.

With few exceptions residual clays, or those derived from the decomposition of rocks in place, are fit only for the commoner grades of brick and earthenware. Nearly all the smoother and finer textured clays are formed from the sedimentary plastic products of rock decay. These beds may be, however, of a widely different nature, some being sandy, or to use a technical expression, "lean," and others very fine-grained, plastic, and "fat." By the accumulation of beds of fine-grained material, one on top of another, the clays become consolidated into shales. These shales, which are much used in certain localities, are sometimes as hard as rock, but when ground and mixed with water yield a plastic, workable mass. In actual pottery practice it is usual to mix several different kinds of clay, thus combining the useful qualities of each. Probably more than 90 per cent of all manufactured articles are moulded of a mixture of at least three clays.

The various sorts of clays have come to be distinguished by different names; thus one hears of stoneware, china, and brick clays; pipe clays, ball clays, fire clays, and kaolins. These distinctions are not very exact, and in most cases merely show the local use to which the clay is put. China clays are usually white or burn white, and when mixed with ball clay, ground felspar, and quartz, are used in the manufacture of porcelain and other forms of white ware. Ball clay is the name given to very fat plastic clays which can be formed and moulded into shape and which will dry out to a mass of high binding power without suffering deformation. Used by themselves ball clays will rarely stand the fire well, but for mixing with good burning clays of low binding power and little or no plasticity they are very necessary. Florida supplies these clays to a considerable extent, but for special reasons they are at present very largely imported from England and Germany. Kaolins are made up of fine particles of the mineral kaolinite which results from the decomposition of felspar. Kaolin, which, chemically speaking, is a silicate of aluminium, is supposed to be the essential ingredient in all clays, and it is not uncommon to read in various publications that kaolin furnishes the active clay base in all plastic clay. Recent researches made in the Division of Tests of the Bureau of Chemistry, however, show that such statements do not express the whole truth of the matter. Fire clays, as their name signifies, are those which do not fuse or melt except at very high temperatures, 3500° F. and over; although these clays are generally used in the manufacture of fire bricks, muffles, and linings for furnaces, they can be used for decorative purposes, as their very stability in the fire makes them easy to burn.

PHYSICAL PROPERTIES OF CLAY.

We may now turn to the consideration of the physical properties of clay, taking them in the following order; (1) Plasticity; (2) binding power (tensile strength); (3) slaking; (4) air shrinkage; (5) firing qualities—(a) fire shrinkage (distortion), (b) fusibility, (c) colour; (6) absorptiveness.

Plasticity.

It is a matter of common observation that the particles of all wet powders to a greater or a less degree cohere. If we take ordinary clean white sea-sand, grind it to an impalpable powder and mix it with a certain amount of water, we shall have a mass just sufficiently coherent to ball together. If, however, we try to mould the wet mass into a form it quickly crumbles and shows itself quite unworkable. From this extreme up to the most plastic of the ball clays which will respond to every touch of the artist's hand and preserve every line of the graver's tool, we find among the fine-grained materials on the earth's surface every grade of variation.

The cause of this wonderful property has been much discussed by students of clay structure. Investigations carried on in the Division of Tests show that the effect is due to the presence of a certain proportion of particles

which actually soften and become sticky under the action of water. It has been noted that purely crystalline particles do not make plastic masses, but there is another kind of matter than that which appears in a crystalline form. This is broadly defined as amorphous substance. Only one kind of amorphous matter, however, is active in producing plasticity. Glass is an amorphous, non-crystalline material, but powdered glass is no more plastic when wet than is sand. The particles which are active in forming coherent masses are highly hydrated, that is, they contain so-called water of combination, which disappears into some sort of porous structure too fine to be seen even under the most powerful microscope. Clays, even when thoroughly dried in the sun and air so that they have every appearance of being dry powders, will sometimes contain as much as 12 or 14 per cent of this combined water. If the clay is heated to a sufficiently high temperature to drive off this water all plasticity and clay likeness is gone. Thus brick dust is no more plastic than sand or glass-powder. The particles have not become crystalline by heating, but the active hydrated particles have been changed into dead amorphous ones. These active particles which become coherent when wet are called *colloid* (which means glue-like), to distinguish them from the inactive crystalline and amorphous grains which are also present in almost every clay.

Binding Power.

It is this property which provides strength in the air-dried clay so that articles fashioned of it can be transported to the kiln without crumbling and breaking. It is a very important quality and the purer clays and kaolins are often deficient in it. The property is tested in the laboratory by moulding briquettes of a special shape, which after drying out are pulled apart in a machine designed for the purpose. The strength of different kinds of clays, as determined in this manner, is given in the following table:—

Tensile Strength of Clays.

	Pounds per sq. inch.
Pure kaolins.	5—20
Common brick clays	30—100
Pottery clays	100—500
Ball clays and other very plastic clays	200—500

It is a curious fact that though high binding power is usually associated with high plasticity, this is not invariably the case. Some of the plastic clays of New Jersey in use in the potteries are of comparatively low binding power. This can be explained, however, by the colloid theory as outlined in the preceding paragraph better than by any other theory as yet advanced. There are various kinds of particles which assume to varying degrees the soft, almost viscous, condition which leads both to plasticity and binding power. In order to illustrate what is meant let us suppose that an inert, non-plastic powder were to be mixed with egg albumen on the one hand and with a glue solution of equal viscosity on the other. Masses of some plasticity would be obtained in both cases, but on drying out the binding power of the glue would be much higher than that of the albumin. The difference in such a case is attributable solely to the kind of cementing material which is present and its individual character. In addition to this it is undoubtedly true that the shape and size of the particles, and the proportional amount of plastic and non-plastic particles present have an important influence on these properties.

Slaking.

The slaking of clays refers to the quality possessed by some of them of rapidly disintegrating and falling to powder when lumps are thrown into water. Such clays are usually less dense; that is to say, bulk for bulk they are less heavy than those which do not possess this property. Clays that slake, if touched to the tongue will adhere to it, sometimes so strongly that it is painful to draw the tongue suddenly away. Both this effort and the slaking are caused by a vigorous indrawing of water into the porous structure

of the particles. In more dense clays this porous structure is already "stuffed" sometimes with water and quite often with other substances both of organic and inorganic origin. The truth of this is readily shown by a very simple experiment. Glycerin is not absorbed into the clay structure rapidly enough to produce slaking; if, therefore, a lump of slaking clay be soaked in glycerin over night it can be transferred to water, in which it will then be found to behave as does any non-slaking clay.

Clays that have been slaked and the powder moulded into masses and air dried do not again disintegrate when wet, as the porous structure is then sufficiently saturated with water to decrease the action. The power to slake readily is often a great advantage, as it enables hard clay masses from the bank to be disintegrated by weathering and thus does away with the necessity for elaborate grinding processes.

Air Shrinkage.

Air shrinkage is that which takes place during the air-drying of a clay mass and is caused by the evaporation of the water, both that which has been used in mixing and a portion of that which the clay contains. As has been explained, very plastic clays possess a greater number of the active hydrated particles with a porous structure, so that we should expect these clays to exhibit a higher shrinkage than lean clays, and this is actually found to be the case. Everyone is familiar with the way glue-like bodies shrink on drying. This shrinkage varies in different clays from 1 to as high as 10 per cent. We now begin to see why the skill of the potter is highly taxed even before his ware goes to the kiln. If he selects a clay which from its plasticity and binding power is all that could be desired, it is probable that its high air shrinkage will cause it to deform and crack (crack) on drying out. Sometimes by very slow and careful drying the danger can be avoided, but usually another leaner material, sometimes even sand itself, is mixed in just such an amount as to reduce the shrinkage without too far lowering the other useful qualities.

Firing Qualities.

Many difficulties must be overcome in order to obtain a clay mixture which will burn well in the kiln and produce the desired result with the utmost economy. Of course the selection of the right kind of kiln and the proper control of the fire is of paramount importance. Such questions cannot be treated in an article of this nature and larger works on the subject must be consulted.

Fire shrinkage.—After a clay mass is thoroughly air-dried it undergoes a further shrinkage on heating as the water of combination already referred to is driven out, and the particles softened by incipient fusion followed by vitrification, draw together and adhere. By incipient fusion is meant the point at which the particles become soft in the fire and by vitrification the point at which they fuse together. Common bricks and earthenware are baked only to this first point and retain an open and porous structure, the bond of incipient fusion being sufficient to form the clay into tough, stone-like materials. Paving bricks and many kinds of stoneware are vitrified.

Fusibility.—It is well known that if pure crystalline substances are heated they become gradually hotter without apparent softening until a certain critical thermometric point is reached, at which the crystals suddenly melt and the mass assumes a liquid condition. If, on the other hand, a non-crystalline, amorphous substance like glass is heated, it begins very soon to soften and continues to grow continually more viscous until it finally flows. In other words, crystalline substances have sharp and definite melting points while amorphous substances have none at all, but soften gradually to the point of flow. As has already been mentioned, there is evidence to show that clays are mixtures of crystalline and amorphous particles in varying proportions, and it may now be further pointed out that, in the fire, clays behave just as we should expect such mixtures to do. If clays had a sharp melting point,

owing to the difficulty of exact control of temperature, the potter would be in continual danger of melting his ware to formless lumps, while on the other hand he could obtain no vitrified bond unless the particles first softened to the point of incipient fusion. As a matter of fact in most clays the point of incipient fusion is lower by from 100° to 500° F. than the point of vitrification, and this point in its turn may be many hundred degrees below the point of deformation and flow. Common brick clays will usually fuse at 2000° F., while many of the better grades of clays will stand 3000° without viscosity or deformation, while fire clays go as high as 3500° and even more. It will be apparent from what has been said that a clay which will stand a considerable increase in temperature above a given point without softening to the point of deformation and flow will be—other things equal—the safest and easiest clay to burn.

We have now to consider briefly the elements which actually determine the fusibility of clay. Quartz is an oxide of silicon formed by the association of two atoms of oxygen, with one atom of the element silicon to form the oxide silica, which is expressed by chemists by the symbol SiO_2 . Silicon is one of a group of elements which always form acid oxides, so called because if heated with the proper amount of certain alkaline or basic oxides like lime, which is an oxide of calcium (CaO), they will combine to form fusible slags which are neutral, showing neither an acid nor an alkaline character. Silica has a very high melting point, and lime an even higher one, so high in fact that it withstands the extreme temperatures of the electric arc. If, however, the two oxides be mixed together the fusing point is lowered. Thus in clays the point of fusion depends upon the amount of fluxing agents present, such as the basic substances, iron oxide, soda, potash, lime, and magnesia; and, although fineness of grain is also an element in determining fusibility, the point of fusion will usually fall with the percentage of these basic substances present. For this reason the common red brick clays which get their colour from iron oxide are, as a rule, of low fusing point.

Colour.—The colour of a clay before it is burned is of little importance except in so far as it serves to indicate the presence of iron. In the kiln, however, except in perfectly white clays which burn white, colour changes invariably take place. Many black clays stained by organic matter burn white, while others burn to reds, buffs, and cream colours. Nothing of importance has ever been accomplished technically in causing a dark burning clay to burn white. It is known that an increase in the amount of lime will produce a paler burn if the colour is due to iron, but, on the other hand, such an admixture has certain deleterious effects, such as lowering the fusing point unequally.

Absorptiveness.

The power that some clays have to rapidly absorb water and other substances has already been discussed. This property is frequently made use of in the arts. In addition to this action certain unctuous varieties of clay which will not usually adhere to the tongue have the power of removing colouring matters from solutions by actually sweeping to the fine particles of matter held in suspension and "sweeping" them away. These clays are known as fullers earth and are much used as clarifying and bleaching agents. They are usually of a greyish-green colour, very soft and greasy to the touch, and if placed on the tongue they appear to melt away.

(To be continued).

Royal Institution.—A General Monthly Meeting of the Members of the Royal Institution was held on the 2nd inst., Sir James Crichton Browne, M.D., F.R.S., Treasurer and Vice-President in the Chair. Messrs. W. A. Adam, W. A. Harper, J. B. Lightfoot, G. A. Moore, M. H. K. Poser, and Professor J. Perry, F.R.S., were elected Members.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, March 15th, 1906.

Prof. R. MELDOLA, F.R.S., President, in the Chair.

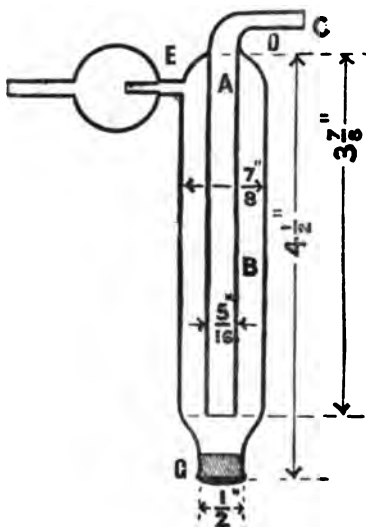
(Concluded from p. 152).

63. "*Aromatic Sulphonium Bases.*" By SAMUEL SMILES and ROBERT LE ROSSIGNOL.

It was shown that the action of thionyl chloride on phenetole in presence of aluminium chloride gives rise to *p*-phenetylsulphoxide and triphenetylsulphonium chloride; the latter is produced by the action of excess of phenetole on the sulphoxide. This reaction has led to the discovery of two other methods of preparing these aromatic sulphonium bases:—(1) From a sulphoxide and phenetole with a dehydrating agent; (2) from a sulphonic acid and phenetole with strong sulphuric acid. It is also shown that in this reaction a sulphoxide is formed as an intermediate product.

64. "*A New Form of Calcium Chloride Tube for Combustion.*" By ARTHUR EDWIN HILL.

The calcium chloride tube indicated in section in the accompanying figure consists essentially of an inner exit tube, A, which is enclosed within a larger inlet tube, B, the latter being fused to the tube A at D, and carrying a side-tube, E, provided with the usual form of bulb in which water can collect as liquid. The apparatus is easily filled by inverting it and pouring finely granulated calcium chloride into both the tubes A and B; before filling, a little glass-wool should be inserted at the top of both the inner and outer tubes, and after charging with calcium chloride more glass-wool is introduced; a suitable cork is then inserted at G; finally, this cork is cut off flush with the bottom of the tube and neatly covered with sealing-wax.



The bulk of the water produced in the combustion is condensed in the bulb on the tube E, the remainder being absorbed during the passage of the moist gases down B and up A. A double scrubbing action is thus obtained by the aid of a comparatively small weight of calcium chloride; this, in fact, in addition to its compactness, is a special feature of the tube and the cause of its efficiency. A certain amount of free space, not packed with the chloride, in the tube B, immediately above the point of connection

with H_2O , is allowed, into which the water vapour can diffuse before coming into contact with the calcium chloride; by this means, the calcium chloride is uniformly wetted.

The new form of tube presents a number of advantages.

1. The double scrubbing action, which ensures complete desiccation by means of a comparatively small quantity of calcium chloride. The whole apparatus when filled weighs only from 25 to 30 grms.

2. It can be very easily emptied and re-filled. There are no tubes to be sealed off at the blowpipe, and only a single cork is required.

3. It is very compact and strong, the bend which is a point of weakness in the ordinary U-tube being got rid of. It is also more easily manipulated than the ordinary form of tube.

4. It can be more easily and rapidly wiped dry before weighing. Several analyses made for the purpose of testing the tube have shown that it gives perfectly satisfactory results. The carbon dioxide produced simultaneously with the water is easily and completely swept from the tube into the potash bulbs by the current of air usually employed in a combustion.

65. "The Viscosity of Liquid Mixtures." (Part III.). By ALBERT ERNEST DUNSTAN.

The author's previous work and his hypotheses regarding maximum and minimum points in viscosity concentration curves were summarised, and some relations between viscosity and molecular weight were indicated.

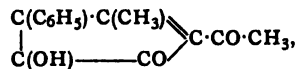
For liquid mixtures of unimolecular components, it was shown that the general viscosity curve is concave to the axis of percentage composition, owing to the reciprocal effect of each liquid on the other.

Moreover, the depression angles which measure these mutual effects are, in general, proportional to the molecular weight of the liquid concerned. The "external angle" lying between the curve and the viscosity axis is shown to be also proportional to the molecular weight of the liquid.

The formulæ proposed by Arrhenius was discussed, and it was pointed out that no formula can interpret the curves which does not contain a factor characteristic of each curve. Each allied substance lies on a simple parabolic curve. The curves connecting molecular weight with the $\log_{10}$ viscosities are straight lines. The associated lower members of each group diverge somewhat from this line. Water, and formic and acetic acids, show anomalous behaviour. If the family to which any liquid belongs is known, the molecular weight of the liquid can be calculated.

66. "The Action of Phenylpropionyl Chloride on the Ketonic Compounds." (Part II.). By SIEGFRIED RUHEMANN.

The compounds previously described by Ruhemann and Merriman (*Trans.*, 1905, lxxvii., 1383) have been subjected to a closer investigation. The red compound $\text{C}_{14}\text{H}_{12}\text{O}_3$, which was represented by the formula—



reacts with phenylhydrazine to yield a phenylhydrazone which, on account of its insolubility in alkali, is considered to have the constitution—



Its formation, therefore, is accompanied by the change of the enolic into the ketonic group. Analogous formulæ should be attributed to the semicarbazone, $\text{C}_{15}\text{H}_{15}\text{O}_3\text{N}_3$, and the oxime, $\text{C}_{14}\text{H}_{13}\text{O}_3\text{N}$. The fact that these two compounds are colourless indicates that the colour of the red compound $\text{C}_{14}\text{H}_{12}\text{O}_3$ is due to the grouping $\text{---CO} \cdot \text{COH} \cdot \text{C} <$,

and disappears with the change into $\text{HC} \cdot \text{CO} \cdot \text{CO}$ —.

The former configuration yields blue salts with alkalis. The results thus obtained are used to establish the structural formulæ of the derivatives of oxalyldibenzylketone (see Claisen and Ewan, *Annalen*, 1895, cclxxxiv., 245), the properties of which resemble the red substance $\text{C}_{14}\text{H}_{12}\text{O}_3$. The former compound with alkalis yields both blue salts and yellow salts. The blue salts owe their formation to the same arrangement of the ketonic and enolic groupings as is contained in the red substance $\text{C}_{14}\text{H}_{12}\text{O}_3$, whilst in the yellow salts the ketonic group occupies the β -position with respect to the enolic group.

PHYSICAL SOCIETY.

Ordinary Meeting, March 23rd, 1906.

Prof. J. PERRY, F.R.S., President, in the Chair.

PROF. F. T. TROUTON read a paper, "On Unilateral Electric Conductivity over Damp Surfaces."

Some time since the author noticed a rather perplexing difference in electrical resistance depending on the direction in which the measuring current was passed. The resistance under examination was that of the layer of moisture which adheres to glass when exposed to moist atmospheric conditions. The arrangement in which this resistance measurement was effected was one used for determining the temperature of deposition of dew. For this purpose two parallel wires of platinum were melted on to a glass surface at a small distance apart. The surface could be artificially cooled. A cell and a galvanometer were inserted in series with the two platinum wires. As soon as moisture condensed on the glass the circuit was completed and a current passed, thus permitting the accurate determination of the dew-point. When a delicate galvanometer is used a small current can be detected long before the true dew-point is reached. It is at this stage that the anomalous behaviour in the resistance is found. On passing a current across the glass surface when exposed to ordinary atmospheric conditions, it was found to diminish to a certain minimum value the amount of which depended on the hygrometric state. On reversal, the current assumed its original value and then diminished to a minimum as before, and so on for further reversals. In order more conveniently to study the matter with larger currents, tin-foil grids were prepared by pasting strips of tin-foil on to glass plates. Every second strip was connected to one pole of the battery while the intermediate strips were connected to the other. In this way was obtained a length of surface lying between parallel strips of tin-foil placed at a short distance apart. Grids of various widths were prepared. One set, consisting of eight glass plates with strips 0.2 c.m. apart, gave a total length between electrodes of 250,000 c.m. When a P.D. of 110 volts was applied, a current of about 9×10^{-4} was found to pass, but gradually this diminished to about 1.5×10^{-4} . On reversal, the current was found to be about 3×10^{-4} , which rapidly rose to about 9×10^{-4} , and then diminished to about 1.5×10^{-4} , and so on for further reversals. The resistance was measured by a Wheatstone bridge when the grid was in a good conducting phase and when in a badly conducting phase, and found to be about 140,000 ohms and 800,000 ohms respectively. That the effects observed were not due to anything similar to ordinary polarisation was shown by the fact that no back-E.M.F. was obtained on short-circuiting through a galvanometer in the usual way, and also by the fact that the ratio of the currents in the two phases was independent of the difference of potential applied. Up to 3000 volts were applied. This would require a back-E.M.F. of 1500 volts to give a falling off in

current to half value. The theory put forward to account for the phenomenon depended on the transportation of moisture over the surface by the current. In this way the effective thickness of the layer might be much diminished by a banking up of the moisture along the edge of one of the metallic electrodes.

Mr. ROLLO APPELYARD expressed his interest in the paper, and pointed out that there were many ways in which electricity could pass between the electrodes. For instance, there was moisture leakage, surface leakage, the electrification effect, and the coherer effect. He suggested that experiments should be made so as to localise the separate effects.

Mr. A. CAMPBELL suggested that experiments might be made with electrodes of silver or platinum deposited electrolytically upon the glass so as to do away with the shellac used to stick down the tin-foil electrodes. He pointed out that moisture had a solvent action upon the glass, and that the film was not one of pure water, but of a solution of salts. He suggested that experiments might be made with films on ebonite or mica.

Mr. W. DUDDALL asked if the effects described might not be due to the formation of a film of insulating oxide along the edge of one of the electrodes. He asked the author what time elapsed between a reversal and the first reading taken after it.

The CHAIRMAN, referring to the author's theory, said that the increase in conductivity on reversal of current was so sudden that he doubted whether the film would have time to spread. Also Mr. Duddall's film would, he thought, not disperse quickly enough.

Prof. TROUTON, referring to the separation of effects, said he had desiccated the glass plates and found them insulators. There was, therefore, no conductivity through the glass. He had used platinum melted on to glass, and got the same effects, so that they could not be due to the shellac used in pasting down the tin-foil. Replying to Mr. Duddall, he said that the time elapsing between a reversal and the first subsequent reading was of the order of a tenth of a second.

A paper on "*The Construction and Use of Oscillation Valves for Rectifying High Frequency Electric Currents*," was read by Prof. J. A. FLEMING.

The author recalled the fact that as far back as 1890, when investigating the Edison effect in glow-lamps, he had shown that the space between the incandescent carbon filament and an insulated metal plate placed in the vacuum bulb possessed a unilateral conductivity, negative electricity being able to pass from the filament to the plate, but not in the opposite direction. This led him to suggest an arrangement of the above kind for separating out or rectifying the oppositely directed currents in an alternating current. This effect was now recognised as due to the copious emission of negative ions or electrons from the incandescent carbon. It was by no means obvious, however, before trial, that any such rectifying arrangement or valve would operate with currents of very high frequency. For example, electrolytic rectifiers such as the aluminium-carbon cell were not available for high frequency currents because a time element entered into the chemical actions involved. In 1904, however, the author discovered that if the carbon filament in an electric glow-lamp was surrounded with a metal cylinder connected to an insulated terminal by a wire sealed through the bulb, and if the filament was made incandescent by an insulated battery, then between the insulated terminal and the negative pole of the battery a unilateral conductivity existed which was operative with currents of any frequency, and the valve so made might be employed to render electrical oscillations measurable by an ordinary sensitive galvanometer. The author exhibited oscillation valves made on this plan, and also showed their uses. If placed in series with a galvanometer, in the circuit of a coil of wire acted upon inductively at a distance by oscillations created in another circuit, the valve gives instructive information as to the damping in the primary

circuit. Thus he showed that when using a carbon-point spark-discharger in the primary oscillation circuit, much larger galvanometer deflections were obtained with very short sparks than with long ones. The author explained that the greatly lessened damping in the short spark caused the mean value of the secondary current to be increased, although the charge put into the primary condenser was diminished. The advantages of using multiple spark-gaps in oscillation circuits was also proved by the use of the valve. The author exhibited a multiple spark-gap discharger made with carbon rods, and claimed for it a much greater constancy of action than a discharger made with metal balls. Experiments were also shown illustrating the manner in which the valve is of use in showing the shielding action of thin metal sheets placed between primary and secondary oscillation circuits. Dr. Fleming also exhibited an experiment showing the copious emission of negative ions from the glower of a Nernst lamp at atmospheric pressure. A Nernst oxide glower was placed horizontally near a brass tube filled with water to keep it cool, and on glowing the oxide rod with a continuous current, it was shown that the space between the hot rod and the cool tube possessed a unilateral conductivity, and would only pass negative electricity in one direction. The author referred to the employment of carbon-filament oscillation-valves such as he had described as wave detectors or cymoscopes in connection with wireless telegraphy. Finally, it was pointed out how, by the use of an electro-dynamometer and galvanometer in series, the amount of rectification produced by the valve could be measured, and it was shown that it might amount to 89 or 90 per cent.

Dr. R. T. GLAZEBROOK expressed his interest in the paper, and hoped that Dr. Fleming would be able in a further communication to give numerical data so that the sensitiveness of the arrangement described could be compared with those of other rectifying devices.

A paper on "*The Use of the Cymometer for the Determination of Resonance Curves*," was read by Mr. G. B. DYKE.

The experiments described in the paper were made with a view to the adaptation of the direct-reading cymometer to the delineation of resonance curves and the determination of the logarithmic decrements of wave trains and the resistance of oscillating sparks. A special form of ammeter was inserted in the cymometer circuit, enabling the current to be observed for any position of the instrument. The ammeter used was of the hot-wire type, with a bismuth-iron junction in contact with the hot wire, the E.M.F. of the junction—and hence the current through the fine wire—being read off from the deflections of a single pivot galvanometer of low resistance. The results of the work of Bjerknes and Drude on the oscillation transformer are referred to, and the application of their final equations to the determination of logarithmic decrements from the resonance curve discussed. Precautions to be taken in the determination of resonance curves in practice are enumerated, and an example is given of the calculations involved in the determination of the logarithmic decrement and the resistance of an oscillatory spark. The double frequency set up in closely coupled oscillation circuits is shown by means of the resonance curve of a wireless telegraph aerial, the mathematical deductions of Oberbeck being thereby confirmed.

Mr. A. CAMPBELL asked the author if he could give an idea of the sensitivity of the hot-wire ammeter. He pointed out that it the instrument was used in a vacuum, the sensitivity was much increased.

Mr. W. DUDDALL said he had tried thermo-junctions in a vacuum and found their sensitivity increased five or ten times. He suggested that the author should replace the bismuth in the junction by constantan.

Mr. DYKE, in reply to Mr. Campbell, said that a current with a maximum value of 0.17 ampere gave a deflection of 80 divisions.

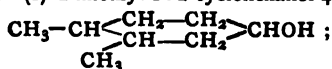
CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

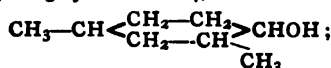
Comptes Rendus Hebdomadaires des Séances de l'Académie des Sciences. Vol. cxlii., No. 10, March 5, 1906.

Sub-oxides of Carbon.—M. Berthelot.—A new volatile suboxide of carbon, having a composition intermediate between CO and CO₂, and of formula C₃O₂, was discovered and isolated by Otto Diets and Bertram Wolf quite recently. It is a liquid boiling at +7°, which spontaneously decomposes. The authors prepare it by the action of phosphoric anhydride on malonic ether, C₃H₄O₄—2H₂O=C₃O₂. The present author observes that it is not correct to attribute the name suboxide of carbon solely to this body, as it represents a whole family of bodies and not a single specimen. He has, in fact, prepared a number of these sub-oxides, and predicts the speedy preparation of many others.

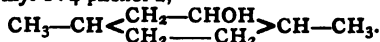
Synthesis of Three Secondary Dimethylcyclohexanols.—Paul Sabatier and A. Mailhe.—The direct hydrogenation of the xlenols allows of the isolation of three secondary dimethylcyclohexanols out of the six possible by theory. Two others had previously been prepared by other methods. The points of ebullition of these three secondary alcohols are 189°, 179°, 178.5°, and are notably higher than those of their tertiary isomers, which are 166°, 169°, and 170°. The composition of the compounds is:—(1) Dimethyl-1.2-cyclohexanol-4,—



(2) dimethyl-1.3-cyclohexanol-4,—



(3) dimethyl-1.4-phenol-2,—



Selenious Anhydride.—Echsner de Coninck.—The author determines the densities of different aqueous solutions of SeO₂. He then investigates the reactions of SeO₂ with HNO₃, H₂SO₄, PCl₅, PCl₃, hydrazine, and hydroxylamine.

Calcium and Strontium Iodomercurates.—A. Duboin.—Already noticed.

Nature of the Decomposition of an Aqueous Solution of Copper Sulphate by certain Aluminium Alloys.—H. Pécheux.—The author investigates the theory of the nature of the decomposition of an aqueous solution of copper sulphate with a small piece of bismuth-aluminium alloy, and proves that the reaction takes place in the following three stages:—

1. $2\text{Al} + 6\text{H}_2\text{O} = \text{Al}_2\text{O}_3 + 3\text{H}_2\text{O} + \text{H}_6.$
2. $\text{H}_2 + \text{SO}_4\text{Cu} = \text{SO}_4\text{H}_2 + \text{Cu}.$
3. $3(\text{SO}_4\text{Cu}) + 2\text{Bi} = (\text{SO}_4)_3\text{Bi}_2 + 3\text{Cu}.$

Estimation of Cadmium.—H. Baubigny.—The author proposes to estimate cadmium by the following processes:—1. Filtration of the precipitate into a weighed funnel of tubular form containing a plug of flax, washing free from acid and calcining in such a manner as to destroy the last trace of organic matter in order to eliminate any chance of loss of cadmium due to reduction. 2. Desiccation of the sulphide in a current of hot air. 3. Treatment by sulphuretted hydrogen gas at a red heat by passing a current of this gas through the filter-tube. 4. Elimination of free sulphur by a current of hot air. 5. Weighing.

Thermo-chemistry of the Hydrazones and the Osazones of α-Diketones and Reducing Sugars.—Ph. Landrieu.—The heats of combustion of a series of

hydrazones and osazones corresponding to the α-diketones and reducing sugars have been determined by means of the calorimetric bomb. The heats of combustion can be used to calculate the quantities of heat which correspond to the fixation of one or two molecules of phenylhydrazine on to the diketones and the sugars.

Benzidine-aniline, Diphenylbidiazoaminobenzene, and Diphenyldisazoaminobenzene.—Leo Vignon.—Benzidine and aniline can be made to unite by two methods—tetrazobenzidine reacting on aniline, or the reaction of diazobenzene on benzidine. An investigation of these reactions, and the formation of diphenylbidiazoaminobenzene, gives an interesting example of the diazo group in the two reactions.

Antimony Tartrate.—J. Bougault.—The use of alcohol should be avoided in the preparation of antimony tartrate. By replacing the alcohol with acetone, a well-defined product is obtained, crystalline and having the formula C₄H₃SbO₆; that is to say, antimony tartrate, C₄H₃SbO₇, less one molecule of water. It is doubtful whether the compound C₄H₃SbO₇ has been obtained in a pure state. Probably all the substances to which this formula is attributed, having been prepared by means of alcohol, ought to contain products of etherification mixed, doubtless, with the compound described in this paper. The differences between M. Guntz's results and those of M. Berthelot on the subject of the heat of solution of Sb₂O₃ in tartaric acid can possibly be explained by the new facts quoted in this paper without employing the hypothesis of two isomeric tartrates, of which latter hypothesis, however, the author does not deny the possibility.

MEETINGS FOR THE WEEK.

TUESDAY, 10th.—Faraday Society, 8. "The Rotating Electric Steel Furnace in the Artillery Construction Works, Turin," by Ernesto Stassano. "Electrothermics of Iron and Steel," by Ch. A. Keller. "Recent Developments in the Gin Electric Steel Furnace," by Gustave Gin. "The Cleaning of Work by means of the Electric Current," by H. S. Coleman.

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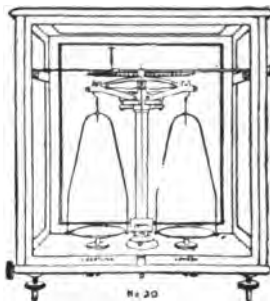
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THE CHEMICAL NEWS.

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NOTES ON THE CLEANING OF WORK BY MEANS OF THE ELECTRIC CURRENT.*

By H. S. COLEMAN.

UNDER certain conditions, work, consisting of iron and brass articles to be nickel-plated, may be cleaned by means of the electric current. Having large quantities to deal with, I could not get sufficient scoured by hand to keep the plating vats fully loaded. After increasing the staff of operators and getting little better results, I was advised to try cleaning by means of the electric current. Cables were carried to the iron tank containing a boiling solution of potassium hydrate (brown Montreal potashes), sp. gr. 1.2° T. A series of experiments were performed, chief of which are given below.

Experiment 1.—Using the iron tank as the cathode, and the work to be cleaned as the anode.

Current flowing for five minutes.

P.D. 1.75 volts.

Result.—Part of the grease appeared to be removed, but the surface of the work (cathode) was covered with a brown-stained deposit, which could not be removed by acid or cyanide dip.

Experiment 2.—Using the iron tank as the anode, and the work the cathode.

Current flowing for five minutes.

P.D. 1.5 volts.

Result.—The work was free from grease and dirt, but the surface was covered with a lead-coloured stain or deposit, which could not be removed by acid dips, and only very slowly by a cyanide dip.

At this stage it was decided to try the effect of reversing the current, in order to remove this "stain" or "deposit," if possible.

Repeating Experiment 2, I waited until the "stain" appeared, and then reversed the current. The result was very satisfactory; the "stain" disappeared, and the surface of the work became clear and bright.

A final experiment was performed to determine the current density required for successful working. This was as follows:—

Approximate area of work ..	8½ sq. ft.
Current	68 ampères.
Working C.D.	8 ampères per sq. ft.
P.D.	2½ volts.

In Experiment 1 the cathode (iron tank) did not show any signs of electrolytic action having taken place. In Experiment 2, however, the anode (iron tank) was attacked, the surface being covered with a thin layer of oxide. This I anticipated from the fact that, during the time the work is the *cathode* the whole of the grease and dirt is removed, the reversal to *anode* being only necessary for the removal of the stain.

That the cleaning is due to an electrolytic, and not merely a chemical change, I feel convinced, for unlike ordinary chemical potash cleaning, the same result is obtained by using a *cold* bath. The cleaning, however, is very much slower than with the hot bath. Electrolysis takes place, and minute quantities of metallic sodium (using sodium hydrate solution only) appear around the cathodes (work), and occasionally take fire. In actual working it is much better to use the hot solution.

By this process the grease, which consists chiefly of tallow, is saponified and remains in solution. The lighter

dirt is thrown upon the surface of the bath and may be skimmed off, the remainder consisting of very fine emery, falling to the bottom of the tank.

A working plant capable of cleaning a considerable quantity of work per day consists of the following, the necessary current being supplied from the plating dynamo:—

There is a wrought iron tank containing the cleaning solution. Over each end is erected a wooden structure, clear of the tank, to support the rods on which the work hangs. Along the top of each support is a flat copper strip, which is connected to the cable by means of a stud. The rods are made of H.C. hard-drawn copper. These make surface contact only, owing to the variety of articles to be cleaned. There is a resistance board and change over switch in the circuit.

In the first stage of the process the iron tank is used as the anode, and the work the cathode. A voltmeter is connected across the terminals of the bath for recording the difference of pressure, the current being recorded on the ammeter placed in the main circuit from the dynamo. The cleaning bath is made up of equal quantities of brown Montreal potash and sodium hydrate dissolved in water and kept boiling, sp. gr. 1.8° T.

The work, after wiring up in the usual way, is distributed along the copper rods in small bunches. The current is now switched on and adjusted until a difference of pressure of 2½ volts is recorded on the voltmeter. Immediately the surface of the work begins to change colour, which takes from 5 to 10 minutes; the current is then reversed for a short time, usually 30 to 40 seconds, until the surface is clear and bright. It is then switched off, and if the work is still dirty the operation is repeated. It is then thoroughly swilled in clean cold water, passed through suitable dipping solutions, electro-coppered, passed through clean, cold water again, and transferred to the plating bath.

The advantages of this process are:—

1. The work is cleaned in one-third of the time occupied by the older method of scouring by hand.
2. It is all wired up ready for plating, and the surface is not touched again by the hand until it is unwired after plating. This not only prevents a certain amount of stripping, but also saves the considerable time which would be occupied in wiring up again, after scouring by hand.
3. The work is rendered *chemically* clean, due to the action of the current working out all grease and dirt from parts where it is impossible to scour with a brush.
4. Time is saved in filling the vats, thus giving an increased output.
5. Saving of labour and materials.

THE USEFUL PROPERTIES OF CLAYS.*

By ALLERTON S. CUSHMAN, Chemist, Division of Tests.

(Concluded from p. 163).

PURIFICATION AND TREATMENT OF CLAYS.

IN ordinary brickmaking the clays are not usually given much preliminary treatment, as it would increase the cost of manufacture too much. In the manufacture of high-grade and art pottery, however, it is often worth while to go to any length in the effort to obtain an even standard of excellence. By means of grinding, washing, and sifting, the clays are reduced to the even and uniform texture which is absolutely essential to the production of high-class work. In addition to these processes it has been found that by allowing the clay masses which have been worked up with water to the consistency of a pasty dough to remain quietly in this condition for long periods, months or more, the binding power and plasticity increase. This

* A Paper read before the Faraday Society, April 10, 1906.

* United States Department of Agriculture, Bureau of Chemistry, Circular No. 17.

is called tempering or ripening the clay, and has been ascribed by some observers to bacterial action. It will readily be seen, however, that according to the colloid theory, as already outlined, the effect gained is caused by the gradual softening of the active particles under the influence of water.

A number of researches have been carried on during the last few years in an attempt to find cheap and economical methods for increasing the useful properties of clays. One experimenter, having occasion in 1901 to seek a clay of the highest possible binding power, soon learned that the material suited to his purpose could only be obtained in Europe, and more particularly in Germany. This experimenter immediately began to seek for a method of treatment for local clays which would develop the useful qualities necessary for his purpose. After some experimentation it was found that by a preliminary soaking in a 2 per cent solution of tannic acid, or the ordinary catechu of commerce, the binding power and plasticity of most clays were very much increased, while the air shrinkage was diminished. Even infusions of dried leaves and straw were found to produce the effect to some extent. The process was called the "Egyptianising of clay," on the somewhat fanciful theory that the ancient Egyptians used straw in brickmaking more on account of the strength given by the infusion than by the fibre.

This subject was investigated in a general way in the Division of Tests, and it was found that many substances have the effect of producing increased binding power and apparently increased plasticity in some clays. This is shown by the tensile strength tests given in the following table, made on a buff-baking brick clay. Each result is the average of 30 to 40 briquettes, from 10 to 12 having been made by each of three operators:—

Average Tensile Strengths of Treated Clays.

	Pounds per square inch.
Water	65
Two per cent solutions of—	
Tannic acid	153
Dilute ammonia	147
Alum solution	98
Iron alum solution	118
Dilute hydrochloric acid	63
Dilute caustic alkali (KOH)	80

Many other substances were also tried, but as the effects were not in all cases decisive they have not been included here. It will be observed that among the solutions tried, tannic acid, ammonia, and the alums were the most active in increasing the tensile strength.

On first thought these results are by no means easy to interpret. Tannic acid and the alums are astringent and of decidedly acid reaction in their water solutions, whereas ammonia is decidedly alkaline in reaction and detergent in its action. On the other hand, if alum is added to clay suspended in water to form a "slip," it will have a flocculating or coagulating effect upon the colloid particles, causing them to settle rapidly. Tannic acid and ammonia appear, in most cases, to have a deflocculating effect, and some clays, suspended in dilute ammonia, show no indication of settling after the lapse of several months. It is known that the colloid structure of clays is selective in its action; that is to say, tannic acid and ammonia will be taken out of solution and retained in the porous particles of a clay. This absorption then probably affects to some extent the flotation of the particles. In the writer's opinion the action of reagents in increasing the binding power of clays is twofold: (1) Deflocculation produces a better distribution of the colloid particles or a so-called "puddling" of the clay; (2) the colloids present are modified and developed, if not actually formed, by the reactions which take place. A further discussion of this interesting subject is of too technical a nature to be given here, but the question will be more fully treated in other publications.

USE OF CLAYS.

The various uses of clays is shown by the following list originally compiled for Mineral Resources, United States, 1891, published by the U.S. Geological Survey ("Clays of the United States East of the Mississippi River," by Heinrich Ries, U.S. Geol. Sur. Professional Paper, No. 11.)

Domestic.—Porcelain, white earthenware, stoneware, yellow ware, and Rockingham ware for table service and cooking; majolica stoves; polishing brick, bath brick, fire kindlers.

Structural.—Brick, common, front, pressed, ornamental, hollow, glazed, adobe, terra cotta; roofing tile; glazed and encaustic tile; drain tile; paving brick; chimney flues; chimney pots; doorknobs; fireproofing; terra cotta lumber; copings; fence posts.

Hygienic.—Urinals, closet bowls, sinks, washtubs, bath tubs, pitchers, sewer pipe, ventilating flues, foundation blocks, vitrified bricks.

Decorative.—Ornamental pottery, terra cotta, majolica, garden furniture, tombstones.

Minor Uses.—Food adulterant; paint fillers; paper filling; electric insulators; pumps; fulling cloth; scouring soap; packing for horses' feet; chemical apparatus; condensing worms; ink bottles; ultramarine manufacture; emery wheels; playing marbles; battery cups; pins, stilt, and spurs for potter's use; shuttle eyes and thread guides; smoking pipes; umbrella stands; pedestals; filter tubes; caster wheels; pump wheels.

Refractory Wares.—Crucibles and other assaying apparatus; gas retorts; fire bricks; glass pots; blocks for tank furnaces; saggars; stove and furnace bricks; blocks for fire boxes; tuyeres; cupola bricks.

Engineering Works.—Puddle; Portland cement; railroad ballast; water conduits; turbine wheels.

THE TESTING AND EXAMINATION OF CLAYS.

Although the testing of clays does not properly fall within the scope of a paper of this nature, a few words on the subject will probably be of interest. There is a very widespread impression that the nature and uses of a clay body will be revealed by a chemical analysis alone. It is true that combined with other data the results of complete chemical analysis are always of value, but the fact remains that clays of widely different properties will have almost identical chemical composition and *vice versa*. This is illustrated by the analysis of two samples, one a fat ball clay from Florida, and the other a rather lean fire clay from Kentucky.

Chemical Analyses of Two Clays.

Constituents.	Florida ball clay. Per cent.	Kentucky fire clay Per cent.
Silica	47.01	47.70
Alumina	38.50	38.47
Iron oxide	0.35	Trace
Lime	0.08	0.112
Magnesia	0.15	0.11
Potash	0.83	0.57
Soda		
Combined water ..	13.78	13.03

In addition to the usual chemical analysis, it is customary to examine clays by methods of so-called *rational* analysis, in which the effort is made to distinguish between the silica which is combined with alumina as clay substance and that which is free as quartz or sand. While somewhat inaccurate such analyses are of undoubted value.

The amount of water required to be mixed with air-dried clays to bring them to just the proper consistency for moulding into forms varies greatly in different clays. It is evident from what has been said in an earlier paragraph that the smaller the amount of water present the less the danger from air shrinkage and the greater the economy of working, especially when artificial heat is used in the drying process. In a thorough report on the useful quali-

ties of a clay this property should always be tested. The physical characteristics which it is necessary to test in a complete examination of clay are best shown by the following laboratory form:—

Physical Characteristics of Clays.

	No. 722 (brick clay).	No. 925 (surface clay).	No. 926 (blue clay).	No. 930 (fire clay).
Water required (per cent) ..	17.4	28.0	19.0	18.5
Tensile strength—				
Average	65	35	130	110
Maximum	72	40	145	115
Air shrinkage (per cent) ..	2	3	7	3
Fire shrinkage (per cent) ..	2	—	8	2
Temp. (°C.) required for—				
Incipient fusion	1140	1000	1140	2500
Vitrification	1430	—*	1430	—
Viscosity	1600	—	1600	—
Colour when burned	Buff	—	Coffee	Cream- whites

* Melted at 1400° to a slag.

When a sample of clay is sent to the laboratory to be tested the following rules should be observed by the shipper. The sample should not weigh less than 30 pounds in its air-dry condition, and it should represent as nearly as possible the average quality of the deposit from which it is taken. The laboratory should be put in full possession of such information as is available both as to the extent of the deposit and the purpose for which the report is desired. In some cases it happens that the interest in a clay is centered in its ability to hold water, for use in building canals or irrigation ditches. Clays vary as much in their permeability as in other qualities, but it is manifestly a waste of the chemist's time to investigate the firing qualities of a clay when simple percolation tests are all that is necessary.

THIRTEENTH ANNUAL REPORT
OF THE COMMITTEE ON ATOMIC WEIGHTS.
DETERMINATIONS PUBLISHED IN 1905.*

By F. W. CLARKE.

THE year 1905 has been remarkably prolific in determinations of atomic weight, and much of the work done was of unusually high quality. Some of the published researches deal with revisions of the more fundamental values, such as the atomic weights of nitrogen, chlorine, iodine, sodium, and potassium. These investigations have made it clear that a general revision and re-calculation of all the atomic weights is now needed, and the developments of the next year or two are likely to bring about many changes in the accepted table. Meanwhile, the International Committee (see Report in CHEMICAL NEWS, xciii., p. 54) advocates a conservative policy, and recommends that changes be deferred until important work now known to be in progress shall have been completed. Then the table of atomic weights, as presented in their report, can be revised intelligently, whereas such a revision undertaken now would be premature. The determinations published during 1905 are given in the following pages.

Nitrogen.

Guye (*Comptes Rendus*, cxl., 1241), from the density and critical constants of nitrous oxide, finds $N = 14.006$. Jaquero and Scheuer (*Comptes Rendus*, cxl., 1384) from densities and compressibilities of several gases, have determined the following molecular and atomic weights:—

$$H = 1.0078.$$

$$NO = 30.005, \text{ whence } N = 14.005.$$

$$NH_3 = 17.014, \text{ whence } N = 13.991.$$

Jaquero and Perrot (*Comptes Rendus*, cxl., 1542), from the density of nitrogen at 1067°, find $N = 14.008$. Guye

* From the *Journal of the American Chemical Society*, xxviii., No. 3.

and Pintza (*Comptes Rendus*, cxli., 51) determined the normal weight per litre of ammonia, nitrous oxide, and carbon dioxide (see under "Carbon," later). The figures for NH_3 and N_2O are as follows:—

NH_3 .	N_2O .
0.77080	1.97762
0.77069	1.97707
0.77073	1.97760
0.77099	
0.77076	

From nitrous oxide, compared with carbon dioxide in corresponding states, $N = 14.006$; from the critical constants, $N = 14.008$. Lord Rayleigh (*Phil. Trans.*, cciv., 369; *Proc. Roy. Soc.*, lxxiv., 446), from the density of nitrous oxide under very small pressures, found $N_2O = 43.996$, whence $N = 13.998$.

Guye and Davila (*Comptes Rendus*, cxli., 826) prepared nitric oxide by three different methods, and determined its density. The weight of one litre at Geneva, under normal conditions, is given below. The three columns relate to the three preparations.

1.	2.	3.
1.3406	1.3403	1.3399
1.3402	1.3398	1.3403
1.3401	1.3400	
1.3407	1.3408	
1.3398	1.3402	
1.3402	1.3402	

If the weight of one litre of oxygen is 1.4290 grms., the ratio $NO : O_2$ gives $NO = 30.012$. Corrected by physico-chemical methods, $N = 14.006$ to 14.010 , according to the method employed. (For the method of correction by critical constants, see Guye, *Journ. Chim. Phys.*, iii., 321.)

Nitric oxide was also the substance chosen by R. W. Gray (*Journ. Chem. Soc.*, lxxvii., 1601) for his determinations of the atomic weight of nitrogen. First, its density was determined in comparison with oxygen, by weighing both gases in the same bulb under corresponding conditions. The following weights were observed at Bonn, where the bulb was filled at 0° and 760 m.m.

NO.	Series I.	O_2 .
0.35845		0.38230
0.35852		0.38229
0.35851		0.38227
0.35849		0.38225
0.35848		0.38226
0.35856		0.38230
	Mean ..	0.38228
	Series II.	
0.35851		
0.35848		
0.35852		
0.35850		

Mean .. 0.35851

Supplementary Determinations.

(Made in London, but corrected to the latitude of Bonn).

0.35848
0.35855

The direct ratio between these weights is $O_2 : NO = 32 : 30.010$, whence $N = 14.010$. Corrected by the method of limiting densities, $N = 14.004$; by the critical constants, 14.008 . Mean value, $N = 14.006$.

Gray effected the gravimetric analysis of nitric oxide by burning finely-divided nickel in the gas. Two sets of determinations were made with nickel from different sources. In the first series, occluded nitrogen in the resultant nickel oxide was determined, and a correction for it was applied. In the second series the occluded gas was

expelled by heating. Only the *corrected* weights are given below.

Weight NO.	Weight O.	Weight N.
	<i>Series I.</i>	
0.31384	0.16729	—
0.64304	0.34300	0.29920
0.50672	0.27025	—
	<i>Series II.</i>	
0.54820	0.29221	—
0.61862	0.32981	0.28885
0.62622	0.33401	0.29234
0.62128	0.33111	—
0.54469	0.29029	0.25432
0.52001	0.27715	0.24270
0.62103	0.33103	0.28998

From these weights the following values for the atomic weight of nitrogen are derived:—

Ratio NO : O ₂ .	Ratio N ₂ : O ₂ .	Ratio NO : N _g .
I. { 14.016 13.996 14.000	— 13.999 —	— 14.001 —
II. { 14.022 14.011 13.998 14.021 14.009 14.020 14.017	— 14.013 14.004 — 14.017 14.011 14.012	— 14.015 14.009 — 14.014 14.003 14.011

Mean of all .. 14.010

The gas remaining after the combustion of the nickel was pure nitrogen, and two density comparisons of it with oxygen were made:—

Weight N ₂ .	Weight O ₂ .
0.32286	0.36889
0.32275	0.36879

Mean .. 0.32280 0.36884

From direct comparison of these figures, $N = 14.003$. Corrected by the method of limiting densities, $N = 14.008$. From the mean of all his determinations Gray puts $N = 14.0085$, or, rounded off, 14.01.

The agreement between all these new values for N , whether gravimetric or physico-chemical, is very striking. In a very complete summary of all the data relative to the atomic weight of nitrogen, Guye has discussed the sources of error in the different methods of determination, and has made it quite clear that the true figure cannot exceed 14.01 (*Bull. Soc. Chim.*, August, 1905, with independent pagination; another paper by Guye is in *Comptes Rendus*, cxl., 1387). The Stas value, 14.04, seems certainly to be high, although the source of error in it remains to be ascertained. The higher value, however, was obtained in two analyses of silver trinitride, AgN_3 , by A. W. Browne (*Journ. Am. Chem. Soc.*, xxvii., 554), who promises further determinations. In Guye's summary, the possibility of a change in the atomic weight of silver is indicated; but that, so far, is only a suggestion. Hinrichs (*Comptes Rendus*, cxl., 1590), in a brief criticism of recent work, argues in favour of $N = 14$.

Chlorine.

Two important memoirs on the atomic weight of chlorine appeared during 1905. First, Dixon and Edgar (*Phil. Trans.*, Series A, ccv., 169) have effected a direct comparison between hydrogen and chlorine. Hydrogen was weighed in palladium. Chlorine, prepared by the electrolysis of fused silver chloride, was weighed in liquid form. The two elements, with chlorine slightly in excess, were made to unite in a glass bulb; the excess of chlorine remaining after union was caused to liberate iodine from a solution of potassium iodide, and then determined by titration with

sodium thiosulphate. The corrected data, reduced to a vacuum standard, are given in the following table:—

Weight H.	Weight Cl.	Atomic Weight Cl.
0.9993	35.1666	35.191
1.0218	35.9621	35.195
0.9960	35.0662	35.207
1.0243	36.0403	35.185
1.0060	35.4144	35.203
0.9887	34.8005	35.198
1.0159	35.7639	35.204
1.1134	39.1736	35.184
1.0132	35.6527	35.188

Mean 35.195 $\pm$ 0.0019

This, of course, refers to hydrogen as unity; if $O = 16$, $Cl = 35.463$. Additional determinations, to include the weight of the hydrochloric acid produced, are promised.

These determinations by Dixon and Edgar have the peculiar merit of directness. Chlorine is referred to one of the fundamental standards, without the intervention of any other element, although the method employed is one of great experimental difficulty, and therefore subject to undiscovered errors. On their face, however, the results are entitled to receive very high weight.

The work of Richards and Wells, on the other hand, is indirect, at least so far as the atomic weight of chlorine is concerned (*Journ. Am. Chem. Soc.*, xxvii., 459; *CHEMICAL NEWS*, xciii., p. 175; original in Publication 28 of the Carnegie Institution). In their research the synthesis of silver chloride was effected by two distinct processes, and from the data so obtained—the atomic weight of silver being assumed—that of chlorine was calculated.

Some of the syntheses were performed by dissolving silver in nitric acid, precipitating as chloride, and weighing the latter in a Gooch crucible. In other cases the nitrate was converted into chloride in a quartz dish, dried therein, and weighed without transfer of material. All weights, of course, were reduced to a vacuum, and many corrections were carefully determined and applied. The corrected results from two series are as follows. (For details, the original memoir must be consulted). The brackets indicate sub-series involving the use of different samples of silver.

Weight Ag. Weight AgCl. Ratio, 100 Ag : AgCl.
Preliminary Series.

9.06483	12.04365	132.861
8.39217	11.14985	132.860
5.37429	7.14056	132.865
8.08222	10.73869	132.868
7.08517	9.41362	132.864
7.97715	10.59837	132.859
8.11978	10.78767	132.857
8.53452	11.33907	132.861
6.73284	8.94511	132.858
8.91366	11.84240	132.857
9.72295	12.91769	132.858
8.63961	11.47862	132.860
11.13795	14.79849	132.865

Mean 132.861

Final Series.

7.24427	9.62508	132.865
8.30502	11.03484	132.870
7.29058	9.68676	132.867
8.58472	11.40614	132.866
8.01318	10.64648	132.862
9.77160	12.98335	132.868
7.98170	10.60528	132.870
11.49983	15.27964	132.868
6.25318	8.30834	132.866
7.72479	10.26360	132.866

Mean 132.867
Stas found .. 132.848

From the final series, if $\text{Ag} = 107.92$, $\text{Cl} = 35.470$. If $\text{Ag} = 107.93$, $\text{Cl} = 35.473$. The authors indicate a preference for the lower value for silver. In either case the value for chlorine is higher than the hitherto accepted 35.455; and its adoption will compel many corrections to be made in the general atomic weight table. The research of Richards and Wells is a model of painstaking accuracy.

From the density and critical constants of hydrochloric acid, Guye (*Comptes Rendus*, cxl., 1241) finds the molecular weight $\text{HCl} = 36.484$, whence $\text{Cl} = 35.476$. A new determination of the density of chlorine gas has lately been published by Treadwell (*Zeit. Anorg. Chem.*, xlvii., 446) but not yet applied to the calculation of its molecular weight.

(To be continued).

A SYSTEM OF QUALITATIVE ANALYSIS INCLUDING NEARLY ALL THE METALLIC ELEMENTS.*

PART I. (continued).—PREPARATION OF THE SOLUTION
(INCLUDING THE DETECTION OF OSMIUM, SILVER,
AND LEAD).

By ARTHUR A. NOYES.

(Continued from p. 160).

Confirmatory Experiments and References.

P. 1, N. 4.—The following minerals or products left a large residue when 1 grm. was treated with HNO_3 (1.20) as above described:—Galenite, PbS ; emery, Al_2O_3 ; chromite, FeCr_2O_4 ; zircon, ZrSiO_4 ; pyrolusite, MnO_2 ; lead dioxide, PbO_2 ; cinnabar, HgS ; fluorite, CaF_2 ; fused silver chloride, AgCl ; and Prussian blue, $\text{Fe}_4(\text{FeC}_6\text{N}_6)_3$. One grm. of fused PbCrO_4 dissolved on three successive digestions with 20 c.c. HNO_3 (1.20). One grm. of precipitated CaSO_4 dissolved upon one treatment with 10 c.c. HNO_3 (1.20), after diluting with 20 c.c. water, and remained in solution upon cooling.

P. 3, N. 1.—In several experiments 500 m.grms. Pb as $\text{Pb}(\text{NO}_3)_2$ were treated with 5 c.c. commercial HF containing some H_2SO_4 . The mixture was then treated according to P. 3 and P. 5. A residue consisting of PbSO_4 always remained, as was proved by decomposing it with Na_2CO_3 and testing for its constituents.

Three c.c. pure 43 per cent HF solution were mixed with 10 c.c. HCl (1.03) in one experiment and with 20 c.c. HCl (1.02) in another; then 1 c.c. 10 per cent $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ solution was added; no precipitate (of BaF_2) separated even on standing ten minutes. One drop (0.04 c.c.) H_2SO_4 (1.20) was then added; a precipitate formed immediately.

P. 3, N. 3.—To 5 c.c. portions of 43 per cent HF solution 1 per cent solutions of various elements were gradually added until a permanent precipitate was produced, waiting two or three minutes after the addition of each half m.grm. or m.grm. The number of m.grms. that had to be added were as follows:—Li, 5; Mg, 2; Ca, 0.5; Sr, 8; Ba, 30; Pb, 0.5; Bi, 0.5; Th, 0.5; Ce, 0.5; Pr, 0.5; Nd, 0.5; La, 0.5; Y, 0.5; and Er, 0.5.

P. 3, N. 4.—Two m.grms. Ta as Ta_2O_5 were heated for fifteen minutes on the water-bath with 5 c.c. 43 per cent HF solution, and the solution was then evaporated to complete dryness, in one experiment twice with the addition of 2 c.c. HNO_3 (1.42), and in another, once without this addition. The residues were then tested for tantalum by the KHF_2 test as described in Part II. In the experiment in which the HNO_3 was added, a satisfactory test was obtained; in the other experiment, no test whatever was obtained, showing that the 2 m.grms. Ta were completely volatilised in the latter case. One m.grm. Ti as H_2TiO_3 was dissolved in 5 c.c. HF , and the solution was then evaporated to complete dryness, in one case twice with the addition of 2 c.c. HNO_3 (1.42), in another once without

that addition. The residues were treated with 20 c.c. H_2O_2 and 2 c.c. HCl . No colour was obtained in the former experiment; a colour corresponding to at least 0.5 m.grm. in the latter.

A mixture of 1 m.grm. Se, 1 m.grm. As, 1 m.grm. Hg, and 1 m.grm. Sb was evaporated to dryness with HNO_3 and treated according to P. 3 (without the addition of SiO_2), and subsequent procedures until the final tests for these four elements were reached. Very satisfactory tests for all of them were obtained, showing they were not volatilised in the HF evaporation.

About 40 m.grms. GeO_2 , made by heating the metal with HNO_3 (1.20) till it was dissolved, evaporating the solution to dryness, and heating the residue at 120° in a platinum dish to a constant weight, were treated with 5 c.c. HF and 5 c.c. HNO_3 (1.42); the solution was then evaporated to dryness, and the residue heated to 120° , and weighed; the change in weight was only 0.1 m.grm.

P. 3, N. 5.—100 m.grms. Ta as Ta_2O_5 were dissolved in 5 c.c. HF and evaporated to dryness three times with 3 c.c. HNO_3 (1.42); the residue was then boiled with 15 c.c. HNO_3 (1.05) for one minute; the solution after filtration, neutralisation with NH_4OH , and addition of $\text{H}_2\text{C}_2\text{H}_3\text{O}_2$ and CaCl_2 gave no precipitate, showing the absence of HF .

The same experiment was repeated with 100 m.grms. Nb as Nb_2O_5 and with 100 m.grms. Ti as H_2TiO_3 with just the same result.

P. 3, N. 6.—Fifty m.grms. Ca as $\text{Ca}(\text{NO}_3)_2$ were digested on a water-bath fifteen minutes with 5 c.c. HF ; 5 c.c. HNO_3 (1.42) were added, and the solution was evaporated to complete dryness, the residue was covered with HNO_3 (1.42), and this was evaporated off; 10 c.c. HNO_3 (1.05) were then added, and the solution was made alkaline with NH_4OH ; only a very slight turbidity resulted.

The same experiment was repeated several times with 200 and 500 m.grms. Ca; in every case a residue remained, or a milky colloidal solution resulted upon treatment with HNO_3 (1.05).

500 m.grms. Pb as $\text{Pb}(\text{NO}_3)_2$ and 500 m.grms. Bi as $\text{Bi}(\text{NO}_3)_3$ were treated in separate experiments in the manner just described; a perfectly clear solution was produced on adding the 10 c.c. HNO_3 (1.05).

A mixture of 250 m.grms. Mg, 250 m.grms. Ba, 250 m.grms. Sr, and 250 m.grms. Li were treated in the same manner; a perfectly clear solution resulted on the addition of the HNO_3 (1.05), and this gave no precipitate on neutralising with NH_4OH , and adding $\text{H}_2\text{C}_2\text{H}_3\text{O}_2$ and CaCl_2 .

P. 3, N. 7.—In regard to the solubility in HF of the oxides of the tungsten and niobium groups, see C. E. on P. 6, N. 1.

P. 3, N. 8.—500 m.grms. Ca, as $\text{Ca}(\text{NO}_3)_2$, were treated according to P. 3 with addition of SiO_2 , and then by P. 5 and 6; no residue whatever remained after the final treatment with HF . This experiment was repeated many times with the same result.

250 m.grms. Y, 250 m.grms. Ce, 500 m.grms. Nd, a mixture of 50 m.grms. Er, and 50 m.grms. Y, and a mixture of 500 m.grms. Nd, and 500 m.grms. La (all as nitrates) were treated in separate experiments according to P. 3, 5, and 6; in all of these cases there was no residue, or only a very small one (of 2–3 m.grms.) after the final treatment with HF .

Portions of 250 m.grms. Th, and of 50 m.grms. Th, as $\text{Th}(\text{NO}_3)_4$, were treated according to P. 3, 5, and 6; on treatment with HF a dense crystalline residue estimated at one-tenth the original amount remained. This experiment was repeated many times with the same result.

P. 3, N. 11.—100 m.grms. Ta, as Ta_2O_5 , were dissolved in 5 c.c. HF , and evaporated to dryness three times with 3 c.c. HNO_3 (1.42); the residue was then boiled with 15 c.c. HNO_3 (1.05) for one minute; after filtration the solution gave a heavy precipitate with NH_4OH . The experiment was repeated with the modification that the residue was heated at 120° for seventy minutes after the

* From the *Technology Quarterly*, xvi., No. 2.

three evaporations; the HNO_3 (1.05) solution then gave no precipitate whatever with NH_4OH . This shows that dehydration is necessary and sufficient to make the Ta_2O_5 insoluble.

The same experiment was twice made with 500 m.grms. Sn; the HNO_3 solution of the residue which was not dehydrated gave a large precipitate with NH_4OH , and with H_2S (after evaporation with HCl).

P. 5, N. 1.—A series of experiments was made in which solutions of various elements were evaporated to dryness with HNO_3 , and the residues were heated in a hot closet for one hour, and then boiled gently with 5 c.c. HNO_3 (1.20) for five minutes; the solutions thus obtained were diluted with 15 c.c. water, heated to boiling, filtered, and evaporated to dryness in crucibles, which were then gently ignited and weighed. It was found in this way that from a residue of 30 m.grms. Sn, as H_2SnO_3 , only 0.3 m.grm. dissolved; from one of 70 m.grms. W, as H_2WO_4 , only 1.1 m.grm. dissolved, which was proved to be not tungsten, but some impurity, by its complete insolubility in NH_4OH ; from a residue of 56 m.grms. Ta, as TaO_5H_5 , only 0.4 m.grm. dissolved; from one of 100 m.grms. Nb a few m.grms. dissolved, but on adding NH_4OH to the solution a precipitate formed which dissolved completely in dilute HCl , proving that no niobium dissolved in the HNO_3 ; from a residue of 22 m.grms. Sb, as Sb_2O_3 , 1 m.grm. Sb dissolved on the first treatment with HNO_3 , and 0.9 m.grm. on each of the two following treatments; and from a residue of 85 m.grms. Ti, as H_2TiO_3 , 8 m.grms. dissolved.

750 m.grms. metallic Sn were treated with HNO_3 (1.42), the acid was evaporated off, and the residue heated at 120° for an hour. The residue was then heated for ten minutes with 5 c.c. HNO_3 (1.20), 20 c.c. water added, the solution heated to boiling, and filtered. The filtrate was evaporated with HCl , and tested for tin by means of the HgCl_2 test. No tin was found to be present.

A mixture of 1 m.grm. Ti, 1 m.grm. Ta, 1 m.grm. Nb, 1 m.grm. W, 1 m.grm. Sn, and 1 m.grm. Sb were treated according to P. 3, 5, and 6, and the subsequent procedures (Part II.) for the analysis of the tungsten and niobium groups. Distinct tests were obtained for all the elements except antimony, showing that their oxides remained in the residue insoluble in HNO_3 . (See also T. A., Part II.).

Two m.grms. Sb, as dehydrated Sb_2O_3 , did not dissolve in 5 c.c. boiling HNO_3 (1.20), but it nearly disappeared upon adding 20 c.c. water and boiling. Five m.grms. Sb, as Sb_2O_3 , treated in this way did not dissolve, but almost all of it dissolved upon a second treatment. Ten m.grms. Sb left a large residue even after two treatments.

Fifty m.grms. W, as Na_2WO_4 , and 150 m.grms. PO_4 , as Na_2HPO_4 , were evaporated with HNO_3 , and heated at 120° for two hours; upon adding HNO_3 (1.20), and diluting, everything dissolved.

The same procedure was followed with a mixture of 5 m.grms. W, as Na_2WO_4 , and 50 m.grms. As, as N_3AsO_4 , with the same result.

Ten m.grms. W were precipitated as H_2WO_4 out of a Na_2WO_4 solution by boiling with HNO_3 , and to the mixture 1 c.c. saturated $\text{H}_2\text{C}_4\text{H}_4\text{O}_6$ solution was added; the precipitate immediately dissolved.

Fifty m.grms. Sb, as Sb_2O_3 , after dehydration at 120° were treated in one experiment with 10 c.c. half-saturated citric acid solution, and in another experiment with 10 c.c. of a mixture of commercial lactic acid with its own volume of water; upon warming, the residue dissolved.

To 1 m.grm. W, 1 m.grm. Nb, 1 m.grm. Ta, 1 m.grm. Sn, 20 m.grms. Sb (in separate experiments) in solution in 3–5 c.c. HF , 250 m.grms. H_3BO_3 , and 5 c.c. HNO_3 (1.42) were added. The solution was evaporated to dryness, and the residue treated with HNO_3 (1.20), which was then diluted with water. In each case, a small residue of the metallic oxide remained, and this did not go into solution on the addition of 500 m.grms. more H_3BO_3 .

To 1 grm. H_3BO_3 , 5 c.c. HF , and 5 c.c. HNO_3 (1.42) were added, and the solution was evaporated to dryness on a water-bath; no residue was left.

A mixture of 500 m.grms. Ti, as TiO_2 , and 500 m.grms. Zr, as $\text{Zr}(\text{NO}_3)_4$, was dissolved in HF , and evaporated to complete dryness four times. It was then heated at 120° for two hours. The residue was treated twice with HNO_3 and water according to the procedure. On the second treatment it went almost entirely into solution, showing that zirconium causes even large quantities of titanium to dissolve.

P. 5, N. 3.—500 m.grms. Mo, as MoO_3 , were dissolved in HF , and following P. 3, the solution was evaporated three times with HNO_3 (1.42), and heated at 120° for an hour. The residue was twice heated with 5 c.c. HNO_3 (1.20), which was afterwards diluted with 20 c.c. water. After the two treatments about one-third of the molybdenum remained undissolved.

The experiment was repeated with 100 m.grms. Mo. A fairly large residue remained after the first treatment of the dehydrated residue with HNO_3 , but only a very small one (2–3 m.grms.) after the second treatment.

In regard to Mo in presence of W or Sn, see T. A., Part II.

A mixture of 200 m.grms. metallic Sb and 50 m.grms. Bi, as $\text{Bi}(\text{NO}_3)_3$, was treated with HNO_3 (1.20), evaporated to dryness, and heated at 120° . The residue was heated ten minutes with 5 c.c. HNO_3 (1.20), 20 c.c. water were added, and the mixture was heated to boiling and filtered. The residue was washed thoroughly with hot water and dissolved in HCl (1.20). To the solution NH_4OH and $(\text{NH}_4)_2\text{S}$ were added. Complete solution took place.

The experiment was repeated with a mixture of 500 m.grms. Sb and 500 m.grms. Bi. Even though the dehydrated residue was twice treated with HNO_3 , and thoroughly washed with HNO_3 (1.05), a large black precipitate of Bi_2S_3 remained after adding NH_4OH and $(\text{NH}_4)_2\text{S}$ to the HCl solution of the HNO_3 residue.

A mixture of 50 m.grms. Se, as SeO_2 , and 200 m.grms. Sb, as SbCl_3 , was evaporated three times with HNO_3 , and the residue heated at 120° . It was then treated with HNO_3 and water in the usual manner, and thoroughly washed with water. The undissolved residue was treated with HF , HCl , and H_2SO_3 . The black precipitate which separated was boiled with HNO_3 , the solution distilled with HBr , and the distillate treated with H_2SO_3 . A red precipitate of Se, amounting to perhaps 5 m.grms., separated.

In regard to ThF_4 , see C. E. on P. 3, N. 8.

GeO_2 was made by heating the metal with HNO_3 (1.42), evaporating with HF and then with HNO_3 , and dehydrating at 120° ; the residue, which then weighed 110 m.grms., was boiled one minute with 5 c.c. HNO_3 (1.20), then 20 c.c. water were added, the mixture was boiled again, and filtered, and the filtrate was evaporated and the residue heated at 120° and weighed; it weighed 89 m.grms., showing that 21 m.grms. had dissolved. This residue of 89 m.grms. was then treated again in the same manner; this time 28.5 m.grms. dissolved.

Fifty m.grms. Se, as SeO_2 , and 200 m.grms. metallic Sn were treated in the same manner, except that the residue insoluble in HNO_3 was dissolved in HBr , the solution distilled, and the distillate treated with H_2SO_3 and KI ; a larger red precipitate of Se (of perhaps 10 m.grms.) was obtained.

Fifty m.grms. Te, as TeO_2 , with 200 m.grms. metallic Sn in one experiment, and with 200 m.grms. Sb, as SbCl_3 , in another, were treated in the same manner, except that the residue insoluble in HNO_3 was dissolved in HF , the solution evaporated with HCl , and treated with H_2SO_3 ; a large black precipitate of Te formed in each case.

In regard to Se and Te in presence of elements of the tungsten and niobium groups (see also T. A., Part II.).

Fifty m.grms. Zr, as $\text{Zr}(\text{NO}_3)_4$, with 600 m.grms. PO_4 , as Na_2HPO_4 , in one experiment, and with 800 m.grms. As, as As_2O_3 , in another, were evaporated with HNO_3 , and the residue heated at 120° . On treatment with HNO_3 (1.20) and dilution, a large residue remained, and the solu-

tion gave a large precipitate with NH_4OH , showing that the zirconium divided itself between the two.

The experiment was repeated with a mixture of 500 m.grms. Zr and 50 m.grms. As; scarcely any residue remained after the treatment with HNO_3 .

The last experiment was repeated, substituting 500 m.grms. Ti dissolved in HF for the 500 m.grms. Zr. The HNO_3 extract of the residue, after evaporation with 1 c.c. H_2SO_4 , gave, upon addition of one-fifth its volume of HCl (1:2) and saturation for one hour at the boiling temperature, a considerable precipitate of As_2S_5 . The residue after similar treatment also yielded a precipitate of As_2S_5 , which was somewhat smaller than that obtained from the solution, showing that arsenic divides itself between the residue and solution when titanium is present.

250 m.grms. Th, as $\text{Th}(\text{NO}_3)_4$, and 250 m.grms. PO_4 , as Na_2HPO_4 , were evaporated with HNO_3 , and heated at 120° for two hours. On heating on a water-bath with 10 c.c. HNO_3 (1:20), everything dissolved except a few hard particles. On adding 20 c.c. cold water a large flocculent precipitate formed, and upon then heating on the steam-bath the solution gelatinised.

Fifty m.grms. V, as $(\text{NH}_4)_3\text{VO}_4$, and 500 m.grms. Sn, as SnCl_2 , were evaporated repeatedly with HNO_3 , and the residue was heated at 120° , and then treated with HNO_3 (1:20). A bright yellow residue remained, which upon treatment with HCl (1:20) yielded a blue-green solution, and the HNO_3 solution was of a brown colour, thus showing that the vanadium was divided.

P. 5, N. 4.—See C. E. on P. 3, N. 3, and T. A. Part II.

P. 5, N. 5.—500 m.grms. Zr, Th, Te, Bi, Al, Cr, Pb, and 75 m.grms. Ga, as nitrates, were, in separate experiments, evaporated to dryness with HNO_3 , and the residue heated for an hour at 120° ; this was then heated with 5 c.c. HNO_3 (1:20), and finally with 20 c.c. water; everything dissolved in this single treatment; the gallium did not do so, however, until the 20 c.c. water were added. The experiment was repeated with 500 m.grms. V, as $(\text{NH}_4)_3\text{VO}_4$, dissolved in HF, and with 500 m.grms. Fe, as FeCl_3 ; two treatments of the dehydrated residue with HNO_3 and water were necessary and sufficient to produce complete solution.

A mixture of 500 m.grms. Ti, as TiO_2 , and 500 m.grms. Zr, as $\text{Zr}(\text{NO}_3)_4$, was dissolved in HCl , and the mixture evaporated three times with the addition of HNO_3 (1:42), and then heated at 120° for two hours. The residue was treated twice according to the procedure with HNO_3 (1:20) and water, and the residue still remaining was dissolved in HF, the solution evaporated with 1 c.c. H_2SO_4 (1:84), and then diluted with 100 c.c. H_2O_2 , and 1 c.c. Na_2HPO_4 was added. No precipitate of $\text{Zr}_3(\text{PO}_4)_4$ separated on twenty minutes' standing.

A mixture of 50 m.grms. each of Al, Ni, V, Cr, Be, and Zr was treated in the manner just described; everything dissolved on the first treatment with HNO_3 .

The C. E. on P. 3, N. 8, made with the rare earth elements other than thorium show that even large quantities of these dissolve completely upon the treatment with the HNO_3 .

Twenty m.grms. Au, as AuCl_3 , were four times evaporated with a little HNO_3 (1:42), and then heated at 120° for two hours. On heating with 5 c.c. HNO_3 (1:20), all but 2—3 m.grms., consisting of a metallic powder, dissolved.

P. 6, N. 1.—500 m.grms. Sn, as metal, of Sb, as metal, of W, as Na_2WO_4 , of Ti, as TiO_2 , of Mo, as MoO_3 , of Te, as TeO_2 , and 30 m.grms. Ge, as GeO_2 , were treated in separate experiments with HNO_3 , the liquid evaporated off, the residue heated at 120° for an hour or more, and then heated on a steam-bath for five minutes with 5—10 c.c. HF; a clear or slightly opalescent solution resulted in every case except that of tin. Twenty c.c. cold water were then added to each solution; the tin solution became clear, and all the others remained so.

To 5 c.c. HF, diluted with 25 c.c. cold water, 1 per cent Bi solution was added, 1 m.grm. Bi at a time,

until a permanent precipitate formed; 6 m.grms. Bi were required.

To a HF solution of 500 m.grms. Sn, as SnO_3H_4 , 400 m.grms. PO_4 , as Na_2HPO_4 , were added, the mixture evaporated to dryness three times with HNO_3 , and the residue heated an hour at 120° ; then it was heated with 5 c.c. HF, and finally 20 c.c. water were added; a large residue of at least 100 m.grms. remained, and this did not dissolve on a second treatment with HF and water.

See also C. E. on P. 6, N. 3, in those cases where the minerals contain the elements of the tungsten and niobium groups.

P. 6, N. 2.—One grm. of each of the following substances was treated according to P. 1, 3, 5, and 6, with the result that a large residue was left on the final treatment with HF:—Galenite, PbS ; cinnabar, HgS ; molybdenite, MoS_2 ; pyrolusite, MnO_2 ; lead dioxide, PbO_2 ; emery, Al_2O_3 ; chromite, FeCr_2O_4 ; cassiterite, SnO_2 ; rutile, TiO_2 ; fused silver chloride, AgCl ; cyanite, Al_2SiO_5 ; tourmaline, $\text{Al}_2\text{O}_3(\text{Fe}, \text{Mn}, \text{Mg})\text{O}(\text{K}, \text{Na}, \text{Li})_2\text{O} \cdot \text{B}_2\text{O}_3 \cdot \text{SiO}_2 \cdot \text{F}$; beryl, $\text{Be}_3\text{Al}_2\text{Si}_6\text{O}_{18}$; zircon, ZrSiO_4 ; monazite, $(\text{Ca}, \text{La}, \text{Pr})_{15}(\text{PO}_4)_{10}\text{Th}_2\text{P}_2\text{O}_9$.

That andalusite and cyanite, Al_2SiO_5 , and tourmaline are not decomposed by HF is stated by Jannasch in his "Gewichtsanalyse," 1897, 231, 233.

P. 6, N. 3.—One grm. of each of the following substances in the form of an impalpable powder was treated according to P. 1, 3, 5, and 6, with the result that no residue, or only a very small one amounting to 1—5 m.grms., was left on the final treatment with HF:—Magnetite, Fe_3O_4 ; menaccanite, $\text{FeTiO}_3 \cdot \text{Fe}_2\text{O}_3$; pyrite, FeS_2 ; pyrrhotite, Fe_7S_8 ; chalcocopyrite, CuFeS_2 ; stibnite, Sb_2S_3 ; niccolite, NiAs ; fluorite, CaF_2 ; cryolite, Na_3AlF_6 ; gypsum, CaSO_4 ; wollastonite, CaSiO_3 ; tremolite, $(\text{Ca}, \text{Mg})\text{SiO}_3$; titanite, CaTiSiO_5 ; orthoclase, $\text{K}_2\text{Al}_2\text{Si}_6\text{O}_{16}$; labradorite, $(\text{Ca}, \text{Na}_2)\text{Al}_2\text{Si}_3\text{O}_{10}$; garnet,—

$(\text{Ca}, \text{Mg}, \text{Mn}, \text{Fe})_3\text{Al}_2\text{Si}_3\text{O}_{12}$;

gadolinite, $(\text{Y}, \text{Ce}, \text{Be}, \text{Fe})_3\text{SiO}_5$; scheelite, CaWO_4 ; columbite, $\text{Fe}(\text{Nb}, \text{Ta})_2\text{O}_6$; samarskite,—

$(\text{Fe}, \text{Y}, \text{VO}_2)_5(\text{Nb}, \text{Ta})_4\text{O}_{15}$;

and the rocks basalt, obsidian, amphibole, and lava.

P. 7, N. 1.—To 500 m.grms. Pb, as PbSO_4 , successive portions of 5 c.c. HCl (1:20) were added, the solution being boiled after each addition, until the solid all went into solution; 30 c.c. sufficed to produce this result. Ninety c.c. water were then gradually added, keeping the solution at the boiling-point; no precipitate was produced. 800 m.grms. Pb, as PbO_2 , dissolved readily in 30 c.c. HCl (1:20), and remained in solution on adding 90 c.c. water.

To 20 m.grms. BaSO_4 30 c.c. HCl (1:20) were added and the liquid boiled a few minutes; complete solution resulted. Thirty c.c. water were added to the solution kept at the boiling-point, no precipitate formed.

The same experiment was repeated with 100 m.grms. AgCl with the same result.

500 m.grms. fused AgCl in the form of shavings were boiled for three or four minutes with successive 30 c.c. portions of HCl (1:20); the solid all dissolved on the third treatment or in 90 c.c. HCl (1:20).

500 m.grms. Sn, as metal, and 400 m.grms. PO_4 , as Na_2HPO_4 , were evaporated with HNO_3 (1:42), and heated at 120° ; the residue was treated twice with HF and water, as described in P. 6, and the large residue still remaining (amounting apparently to 200 m.grms.) was heated on a steam-bath for ten minutes with 15 c.c. HCl (1:20); complete solution occurred.

P. 7, N. 3.—The following substances, in the form of a fine powder, were submitted, if non-metallic, in quantity of 1 grm. to P. 1, 3, 5, 6, and 7; if metallic, in quantity of 0.5 m.grm. to P. 4, 5, 6, and 7, with the result that a large residue, amounting to at least one-half of the original substance, remained in every case:—Molybdenite, MoS_2 ; emery, Al_2O_3 ; chromite, FeCr_2O_4 ; cassiterite, SnO_2 ; rutile, TiO_2 ; thorium fluoride, ThF_4 ; Prussian blue, $\text{Fe}_4(\text{FeC}_6\text{N}_6)_3$; cyanite, Al_2SiO_5 ; tourmaline; zircon,

ZrSiO₄; monazite, mainly Th₃(PO₄)₄; metallic rhodium, Rh; metallic iridium, Ir; Ural Mts. platinum ore, Pt, Pd, Os, Ir; Ural Mts. osmium-iridium, Os, Ir; crude ruthenium, Ru; ferrochrome, Cr, Fe; ferrosilicon, Fe with 13 per cent Si.

P. 8, N. 1.—Barium sulphate, tourmaline, cyanite, and allied silicates are analysed quantitatively by decomposition with Na₂CO₃. (See Classen, "Quantitative Analysis," Harriman's translation, pp. 62, 204).

330 m.grms. of cyanite, constituting the residue left after treatment of 1 grm. of the mineral by P. 1, 3, 5, and 6, were fused with 7 grms. Na₂CO₃ (this large quantity being necessary to produce a fluid fusion); on treatment first with hot water, and then with HCl, everything dissolved.

One grm. of each of the following substances as an impalpable powder was treated by P. 1, 3, 5, 6, and 7, and the residue was fused with Na₂CO₃ as directed in P. 9:—Emery, Al₂O₃; rutile, TiO₂; cassiterite, SnO₂; monazite, Th₃(PO₄)₄, &c.; and zircon, ZrSiO₄. A large residue was left upon extracting the fusion with water and adding HCl to the residue (P. 10, 11).

The residues undecomposed by Na₂CO₃, obtained in the preceding experiment, were fused with K₂S₂O₇ by P. 12; emery, rutile, zircon, and monazite went completely into solution on adding the fusion to water, or upon treating the residue with H₂SO₄ (1:20). In the case of cassiterite, a large residue (450 m.grms.) was left undissolved.

P. 8, N. 2.—500 m.grms. of each of the following substances, reduced to a fine but far from impalpable powder, were first treated for from ten to thirty minutes with strong aqua regia, and the residues were then introduced into a nickel crucible containing 5 grms. Na₂O₂ in a state of fusion, and the fusion was maintained for from five to twenty minutes;—Molybdenite, MoS₂; carborundum, SiC; ferrosilicon, Fe with 13 per cent Si; ferrochrome, FeCr; Ural Mts. platinum ore, Ural Mts. osmium-iridium, pure metallic iridium, crude metallic ruthenium, and (100 m.grms.) pure metallic rhodium. All of these substances except the platinum metals reacted violently with the Na₂O₂, with the production of much light and heat. Complete solution resulted in case of all the substances upon adding to the fused mass 75 c.c. water, neutralising with HCl, adding a moderate excess of it and heating (except that in case of carborundum a small flocculent precipitate of SiO₄H₄ separated).

P. 12, N. 1.—One grm. monazite was treated with HF and HNO₃ (P. 2 and 5), and the residue was fused with K₂S₂O₇ and H₂SO₄ (P. 12). On adding the mass to water, a flocculent precipitate formed; but this dissolved on decanting the liquid, and adding 10 c.c. H₂SO₄ (1:20).

500 m.grms. Ti, as TiO₂, were fused with 5 grms. K₂S₂O₇ and 4 c.c. H₂SO₄ were added. On adding the mass to 50 c.c. water everything dissolved, and remained in solution upon boiling.

Ten grms. K₂S₂O₇ were heated at a dull red heat in a covered platinum crucible for twenty minutes. The mass was cooled, 2 c.c. H₂SO₄ (1:84) were added, and the mixture heated for fifteen minutes below its boiling-point till a clear fusion resulted. On cooling, about half the mass separated as a compact solid, and the other half remained viscous. On adding 2 c.c. more of H₂SO₄ (1:84), re-heating for ten minutes till a clear fusion was obtained, and then cooling, the mass became viscous, but was so liquid that it could be poured out.

P. 12, N. 3.—The liquid mass obtained in the last experiment was added to 30 c.c. water, two drops HCl (1:20) added, and the solution was then shaken with 1 c.c. Ag. From the colour of the precipitate it was estimated by comparison with the colours obtained with known amounts of platinum, that 2 m.grms. Pt had been taken up from the crucible.

P. 13 N. 1.—See C. E. on P. 8, N. 2, in regard to action of Na₂O₂ on the substances that may be present in a dark coloured or metallic residue.

P. 13, N. 2.—Five grms. Na₂O₂ were fused at a lower red heat for twenty minutes in a 35 c.c. nickel crucible

weighing 17 grms. The crucible, after washing out and drying, had lost 1 grm.

Two grms. Na₂O₂ were fused for twenty minutes on a silver crucible cover; the cover lost 0.9 grm.

In regard to the action of Na₂O₂ on Ni, Fe, Ag, and Pt, see also Dudley (*Am. Chem. Journ.*, 1902, xxviii., 59–66), and Leidié and Quenessen (*Bull. Soc. Chim.*, 1902, [3], xxvii., 179–183).

P. 13, N. 3.—Five grms. Na₂O₂ from Roessler and Hasslacher, N.Y., made from metallic sodium under the Castner patent, was submitted to a qualitative analysis. This quantity contained only about 3 m.grms. Fe and 1 m.grm. Sb as metallic impurities.

P. 13, N. 4.—For the composition of the hydrated oxide of nickel produced, see Dudley (*Journ. Am. Chem. Soc.*, 1896, xviii., 901–902).

P. 13, N. 5.—For the composition of the fusion products of platinum, see Dudley (*Am. Chem. Journ.*, 1902, xxviii., 59); of iridium, Claus (*Z. Prakt. Chem.*, 1846, xxxix., 101).

500 m.grms. Ir, as metal, and 500 m.grms. Pt, as metal, were in separate experiments fused in a nickel crucible with 5 grms. Na₂O₂ for seventy-five minutes. The fused mass was treated with 100 c.c. water, and the coal-black residue allowed to settle. The very slightly yellowish solution was decanted, acidified with HCl, and evaporated nearly to dryness; the residue was treated with HCl (1:20); the solution was decanted from the pure white NaCl, and evaporated to dryness with HNO₃; the residue was dissolved in 1 c.c. water, and the solution was saturated with NH₄Cl. In the case of the iridium, only about 1 m.grm. Ir separated as a black precipitate, showing that the fusion product was almost insoluble in water. This is in disagreement with the statement of Leidié and Quenessen (*Bull. Soc. Chim.*, 1902, [3], xxvii., 181), according to which the iridium after fusion dissolves completely in water with production of a deep blue solution. In the case of the platinum, no (NH₄)₂PtCl₆ was obtained. The residue insoluble in water was thoroughly washed, and it was then warmed with HCl (1:20). In the case of the iridium it very quickly dissolved to a clear dark blue solution, which during evaporation changed to a dark red colour. In the case of platinum, a large quantity of a dense yellow residue remained after long boiling with HCl (1:20) and strong aqua regia. The HCl solution, however, contained a large quantity of platinum, as was shown by precipitating it in a pressure bottle with H₂S.

500 m.grms. crude Ru were fused with 5 grms. Na₂O₂, and treated with water in the usual manner. The aqueous solution was dark orange-red, and upon adding HCl to the solution and residue, a clear, very dark red solution resulted.

100 m.grms. commercial Rh were fused with Na₂O₂, and treated with water. The supernatant liquid after the precipitate settled was dark orange-yellow, and it remained so after decanting and acidifying with HCl, but it became colourless on boiling; these results being perhaps attributable to the presence of osmium. The black residue and adhering solution were treated with HCl; in the cold there was a large finely divided cream-coloured residue, but upon heating this almost entirely dissolved, yielding a dark yellow-orange solution.

P. 13, N. 7.—200 m.grms. Os were fused with Na₂O₂, the fused mass treated with water and HCl, and the liquid distilled (as in P. 13); after distilling five minutes, the dark orange distillate was replaced by fresh NaOH; after distilling three minutes longer, a yellow distillate corresponding in colour to that given by 4–5 m.grms. was obtained; on again replacing the distillate and boiling for two or three minutes longer, a colourless distillate giving no precipitate with Na₂S₂O₃, and HCl resulted. Twenty-five c.c. HNO₃ (1:42) were then added to the residual solution, and the distillation continued for ten minutes; the light yellow distillate was then treated with HCl and 2 c.c. saturated Na₂S₂O₃ solution; the precipitate obtained was pure white, showing that the osmium distilled over completely in less than ten minutes, and that the addition of HNO₃ is unnecessary.

P. 13, N. 8.—500 m.grms. crude Ru were fused with Na_2O_2 , the fused mass treated with water and HCl , and the liquid distilled (as in P. 13); a weakly coloured distillate, giving with HCl and $\text{Na}_2\text{S}_2\text{O}_3$ a small precipitate corresponding to 1–2 m.grms. was obtained; this was probably due to a little osmium present in the material used. On further distillation into a new portion of NaOH , the latter remained colourless, showing that the ruthenium does not distil over.

P. 13, N. 10.—In one experiment 200 m.grms. very finely powdered osmium, and in another 500 m.grms. powdered osmium iridium were boiled in a distilling flask with strong aqua regia, and the distillate caught in NaOH solution; in neither case did the distillate give a dark coloured precipitate when tested with $\text{Na}_2\text{S}_2\text{O}_3$ and HCl .

P. 14, N. 1.—That the distillation and test with $\text{Na}_2\text{S}_2\text{O}_3$ will show 0.5 m.grm. Os was proved by experiment.

The distillate obtained by distilling 1 m.grm Ru with dilute H_2SO_4 and KMnO_4 was tested with $\text{Na}_2\text{S}_2\text{O}_3$ by P. 14. A black precipitate was produced.

P. 16, N. 2.—The dense yellow residue of Pt_2O_3 insoluble in HCl and aqua regia, referred to in C. E. on P. 13, N. 5, was allowed to stand in the cold over night under water saturated with H_2S ; it was completely converted to a black sulphide, which dissolved readily in aqua regia.

P. 18, N. 1.—As to the solubility of AgCl in NH_4OH , see Bodländer and Fittig (*Zeit. Phys. Chem.*, 1901, xxix., 602), Whitney and Melcher (*Journ. Am. Chem. Soc.*, 1903, xxv., 78).

500 m.grms. Pb, as PbSO_4 , were boiled with 20 c.c. NH_4OH (0.90), the mixture filtered hot, the filtrate nearly neutralised with HNO_3 , and then acidified slightly with $\text{HC}_2\text{H}_3\text{O}_2$, and a few drops of K_2CrO_4 added; a considerable yellow precipitate of PbCrO_4 formed. The experiment was repeated with the modification that the NH_4OH solution was cooled before filtering; in this case a precipitate equivalent to only about 0.2 m.grm. Pb resulted.

P. 19, N. 1.—The statement as to the quantity of PbSO_4 dissolved by 15 c.c. $\text{NH}_4\text{C}_2\text{H}_3\text{O}_2$ solution is based on a direct experiment.

A mixture of 500 m.grms. Ba and 500 m.grms. Sr, freshly precipitated as BaSO_4 and SrSO_4 , was collected on a filter, a portion of 10 c.c. hot $\text{NH}_4\text{C}_2\text{H}_3\text{O}_2$ reagent was poured several times through the filter, the filtrate was acidified with $\text{HC}_2\text{H}_3\text{O}_2$, and a few drops of K_2CrO_4 were added; no precipitate resulted.

500 m.grms. Sr, as SrSO_4 , were boiled with 20 c.c. of the $\text{NH}_4\text{C}_2\text{H}_3\text{O}_2$ reagent, filtered while hot, and 30 c.c. of $(\text{NH}_4)_2\text{CO}_3$ added; after standing for ten minutes, a precipitate estimated at 3–5 m.grms. had separated.

For the solubility of SrSO_4 in HCl (1.04) and HNO_3 (1.032) see Fresenius ("Quantitative Analysis," Am. Ed. of 1881, p. 815).

P. 20, N. 1.—500 m.grms. Sr, as freshly precipitated SrSO_4 , were boiled for ten minutes with 10 c.c. saturated Na_2CO_3 solution, the precipitate washed, and then treated with 10 c.c. cold HCl (1.12); it entirely dissolved.

500 m.grms. Ba, as freshly precipitated BaSO_4 , were boiled for five minutes with 25 c.c. saturated Na_2CO_3 solution; the solution was then decanted and the residue boiled for five minutes, with 10 c.c. Na_2CO_3 solution; the solution was again decanted, the residue washed and treated with 10 c.c. HCl (1.02); it dissolved completely.

P. 20, N. 2.—For the theory of the equilibrium between two difficultly soluble salts in the presence of soluble ones with common ions and the results in the case of BaSO_4 and BaCO_3 , see Nernst ("Theoretische Chemie," 3rd Ed., p. 498).

As to the solubility of BaSO_4 , BaCO_3 , SrSO_4 , and SrCO_3 in water, see Kohlrausch and Rose (*Zeit. Phys. Chem.*, 1893, xii., 241). It should not be forgotten, however, that the values given in the cases of the carbonates are much higher than correspond to the true solubility products, owing to the effect of hydrolysis.

(To be continued).

A REVISION OF THE ATOMIC WEIGHTS OF SODIUM AND CHLORINE.*

By THEODORE WILLIAM RICHARDS
and
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INTRODUCTION.

THE investigation of the atomic weights of the extremely common elements sodium and chlorine was undertaken because of an unaccountable discrepancy which appeared in the composition of a sample of very pure sodic bromide, as compared with the results of Stas. This sodic bromide had been prepared for the purpose of determining the transition temperature of its hydrated crystals; and a preparation which yielded a constant transition point, therefore giving evidence of great purity, nevertheless possessed a perceptibly lower combining weight than that indicated by Stas's results.

Such a discrepancy as this was not to be passed over lightly. It indicated either an unknown constant impurity in our sodic bromide—and hence a possible error in our transition temperature—or else a flaw in the classical work of Stas. When the first cause of disagreement had been carefully sought in vain, the second was pursued.

Thus a physico-chemical investigation demanding great purity of materials led to a quantitative research of unexpected magnitude; and in turn this quantitative investigation depended continually upon physico-chemical methods and considerations. In confirmation of previous work at Harvard, it was found that the physico-chemical conditions of experiment were of as serious import as the purity of the materials, and of far greater significance than an increase in the scale of operations. The essential circumstances here defined must receive consideration in any other research of a similar kind.

It is needless to say that an investigation seeking to test the accuracy of Stas's work is a serious undertaking, for undoubtedly much of his work deserves a rank among the most accurate of quantitative chemical results; certainly he distanced all those who preceded him. Nevertheless, he was by no means infallible, as his long oversight of the solubility of argentic chloride, his difficulty in obtaining colourless ammoniac bromide, the uncertainty concerning the oxygen in his silver, and the recent proof of an important error in his work on iodine,† all testify. Moreover, Stas's work is not to be considered as accurate in proportion to the scale of his experiments, as many have been inclined to suppose. Mere increase in quantity of material can avail nothing if the same physico-chemical error or the same chemical impurity contaminates the whole. Indeed, it is doubtful if beyond a moderate scale of operations increase of quantity decreases even the probability of accidental error. For example, the extreme variations in his work on the synthesis of argentic chloride, in which on the average nearly 200 grms. of the compound were weighed in each analysis, amounted to 0.006 per cent, or 12 m.grms. of material (J. S. Stas, "Oeuvres complètes," Brussels, 1894, i., 341). An accuracy of 0.6 m.grm. with 10 grms. of substance, very easy to attain as far as the collection and weighing of the material is concerned, would have brought him as good agreement. But large quantities have no advantage to offer besides the increased accuracy in weighing and the diminished percentage effect of a given accidental loss of material in transference. For this reason the use of extremely large quantities must rather be considered as evidence of a lack of sound judgment than an index of unusual accuracy. Stas himself acknowledged this by using only about 10 grms. of material in each of his last experiments ("Oeuvres," iii., 503).

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† The work of Ladenburg and Scott has been confirmed by Dr. G. P. Baxter, of Harvard University, whose recent analyses show that the atomic weight of iodine can hardly be lower than 126.97. *Proc. Acad.*, 1904, xl., 419.

Regrettably one must admit that a critical study of the work of Stas upon sodium in particular reveals several possibilities of error, as well as at least one inconsistency. He analysed both the chloride and the bromide; of these salts the former received the greater attention, and will be discussed here in greater detail.

In reviewing his results upon sodic chloride there are two series to be considered, separated by an interval of time of over twenty years. Although at first sight these two series seem to have yielded almost identical results, as a matter of fact they are essentially inconsistent with one another. Both results were obtained by the method of Gay-Lussac, titrating weighed amounts of silver in solution with salt; but at the earlier time the latter was added until all precipitation ceased, while at the later time equivalence was assumed when the supernatant liquid gave equal opalescence with excess of either chloride or silver.

Anyone with experience will recognise that a much higher apparent atomic weight of sodium was to be calculated by the first procedure than by the second, other conditions being the same, and yet the results were almost identical. The immediate inference is, of course, that some other condition or conditions must have varied in order to compensate for the effect of the altered method of titration. This inconsistency, which appears also in his work on potassium and ammonium, has never been explained, and one of the functions of the present work was to account for it.

Among the possible sources of error in Stas's work, the most serious seems to have been the practice of dropping solid salt into the solution of argentic nitrate ("Oeuvres," i., 759; also iii., 479). Of course this practice caused the greater part of the argentic chloride to be precipitated in the presence of a concentrated solution of salt, immediately around the solid—a circumstance tending greatly to promote the occlusion or inclusion of sodic chloride in the precipitate. Stas, in his early work especially, was fully awake to the danger of the occlusion of *argentic nitrate* in this precipitate ("Oeuvres," i., 337); but he seems to have had no fear of occlusion of other salts. In the sequel we shall show that the chloride of silver tends to carry down with it many salts from their concentrated solution, and that the details of treatment determine whether or not these impurities may be removed by washing.

Another possible cause of the discrepancy is to be found in the methods used for preparing the materials. In Stas's earlier investigation, where the analytical end-point was erroneous, greater care was taken in preparing the sodic chloride than in the later experiments. In general, when purifying this salt, he employed violent treatment, and very rarely re-crystallised or in any way fractioned his material afterwards. His favourite method was fusion with ammoniac chloride and ammoniac chloroplatinate. Presumably the ammoniac chloride was used to expel other halogens, and the deposited platinum, Stas believed, carried down silica and alumina with it. Except for these possible advantages, this admixture of foreign materials seems to be a doubtful expedient. In the ten experiments of 1860 ("Oeuvres," i., 365), six different preparations were made, from the following sources:—

1. Sodic carbonate neutralised with hydrochloric acid.
2. Rock salt re-crystallised.
3. Sodic sulphate repeatedly fused with ammoniac chloride.
4. Sodic tartrate re-crystallised and fused with ammoniac chloride.
5. Sodic nitrate, similarly treated.
6. Sodic chloroplatinate re-crystallised and fused.

The final step in every case consisted, as usual, in mixing the salt with ammoniac chloride and ammoniac chloroplatinate, fusing until colourless, and decanting the fused mass from the residue. The ten results agreed almost exactly, indicating that the preparations were, at least, identical; but, of course, the results are all in error, because of the erroneous end-point.

Only two preparations were used in the four experiments of 1881 ("Oeuvres," i., 755). For one of these, impure sodic bicarbonate was neutralised with hydrochloric acid. The salt, after three recrystallisations, was repeatedly fused with ammoniac chloride and chloroplatinate ("Oeuvres," i., 685). By evaporating a solution of the resulting salt with chloroplatinic acid, it was converted into sodic chloroplatinate without fractionation of any kind. About nine-tenths of this material was dissolved in water. Stas assumed that any potassic salt would not then dissolve; but this possibility is by no means excluded. The first lot of crystals from the solution was decomposed and fused, and the resulting sodic chloride used in the first two experiments. A second and third lot were also obtained from the mother-liquor of the first lot. The salt made from the third lot was employed in the fourth experiment of 1881. Thus there were only four crystallisations between the starting point (which was admittedly impure) and the final product; and the first three of these crystallisations were of common salt, isomorphous with potassic chloride. In the third experiment of 1881, the salt was made by neutralising pure acid sodic carbonate and repeatedly fusing with ammoniac chloride in platinum, with no fractionation at all. These specimens were probably less pure than the earlier ones.

Thus it appears that every preparation of Stas was fused in contact with platinum, which was usually in a finely divided "nascent" condition, or at least in the presence of decomposing ammoniac chloride. The question is therefore important whether or not platinum can dissolve in melted salt at the temperature of 800°. We have found that fused salt when pure has but a very slight dissolving effect upon platinum, but in the presence of ammoniac chloride or hydrochloric acid at a red heat in the presence of air a noticeable quantity of platinum is dissolved, as is indicated by the loss of weight of the containing crucible. It seems likely, then, that Stas's material contained traces of platinum, either in a very finely divided state or possibly as a platinum salt; but the amount could hardly have been large. Stas invariably found non-volatile residues in his preparations, consisting of silicates of sodium and calcium, which he discovered by vaporising the salt. These residues usually amounted to about 0.004 per cent.

It is not impossible, however, that yet other impurities might have volatilised with the sodic chloride. In volatilising his salts, Stas used a heat of pure platinum ("Oeuvres," i., 681). It is possible that pure platinum would itself have volatilised appreciably at the temperature required to drive off 10 grms. of salt in half-an-hour.* For either of the two reasons just given, Stas's correction for impurity in his salt would not have been large enough.

These impurities must, however, have been almost equally present in the early and later samples, and could not explain the serious discrepancy between them. At most, however, as we shall show, they could hardly have exceeded 0.01 per cent, an amount much smaller than the discrepancy in question. Our own experience shows that common salt is a substance very easy to prepare in a pure state. Clearly the cause of the discrepancy must be sought elsewhere.

The other essential substance, to be prepared in a pure state, was silver. This Stas prepared in a variety of ways, as is well known; but for all this work the silver was either cast in ingots from under an oxidising flux, or else "granulated" by dropping into water. His criterion of pure silver was the melting of the surface of a button without any apparent irregular expansion as it liquefied, or any flame-colour, and the absence of all specks and spots from the completely melted globule. In all experiments it was reddened in a silver crucible before weighing.

After Dumas had recommended the fusion of silver in a

* See Hall (*Journ. Am. Chem. Soc.*, 1900, xxii., 494); Richards and Archibald (*Proc. Am. Acad.*, 1903, xxxviii., 460); Hulet and Berger (*Journ. Am. Chem. Soc.*, 1904, xxvi., 1512). There seems to be no doubt that platinum is slightly volatile at 1000° in the presence of air. Probably it is not in pure nitrogen.

vacuum, Stas made many experiments upon the occlusion of gases by silver. He carefully investigated the samples of silver used in his atomic weight researches, and also new preparations. He found that fusion in a flux of sodic nitrate introduced a quantity of oxygen, and that bars and blocks were slightly purer than "granulated" silver. In no case was the amount of retained gas found by Stas enough to affect seriously even a very accurate analysis; but as will be shown later, it is probable that he did not find all the oxygen present. An error in Stas's silver would not account for the discrepancy between his results on sodium and ours; for an impurity in the silver would cause his atomic weight of sodium to appear too low, and not too high. On the other hand, the impurity in Stas's silver is undoubtedly the cause of the difference between his atomic weight of chlorine and ours.

From these considerations it was clear that the reason for the incompatibility of the results on sodium was probably to be sought chiefly in the method of analysis rather than in the impurity of the materials. The long-continued search for the causes of discrepancy led finally to a satisfactory explanation of the whole anomaly. In order to make this clear, the details of our own investigation must be recounted.

BALANCE AND WEIGHING.

The Troemner balance which has served in many similar researches was used during most of the present work (*Proc. Am. Acad.*, 1891, xxvi., 242). Successive weighings on it of the same object rarely differed as much as 0.03 mgrm. For a few of the conclusive final experiments a yet more sensitive and perfect balance, especially made for this kind of work by the same maker, was used.

The Sartorius weights were standardised from time to time by the method devised by one of us (Richards, *Journ. Am. Chem. Soc.*, 1900, xxii., 144). It was found as usual that the larger gold-plated and platinum-plated brass weights very slightly changed from time to time, although the smaller platinum weights did not. All weighings were made by substitution. Furthermore, the major portion of the substituted tare consisted of a counterpoise exactly similar to the crucible, tube, or boat which was being weighed. Thus the weights required were never large in amount, and errors due to changes in meteorological conditions were avoided. The method of recording and checking the weights is given in another place (Richards, *Proc. Am. Acad.*, 1895, xxxi., 175; *Zeit. Anorg. Chem.*, 1895, x., 19).

While these obvious particulars are given here for the sake of completeness, it must be remembered that accuracy of weighing is easily attained. The great errors in chemical quantitative work are usually not in the weighing, but are due rather to impurities in the substances weighed or incompleteness or irregularity of reaction. Only in a few cases, with large vessels, was it necessary to use a telescope for reading the vibrations, the observer being in another room and looking through an intervening glass door.

The balance-room devoted to these most accurate determinations was kept at a very constant temperature, being wholly inside a private laboratory whose air temperature was regulated by a delicate thermostatic attachment to its heating apparatus. The balance-room itself had neither heating apparatus nor outside windows. This question of constancy of temperature is of the most vital importance in the weighing of large vessels.

In reducing weights in air to weights in vacuum, the densities at about 20° were assumed to be as given below. The value for silver proceeds from Stas, and was checked by one determination by us of the purest silver (sample W, described later). The value for the brass weights refers to the gold-plated Sartorius weights used in all but a very few of the experiments; the other set had a slightly higher density, of which account was taken. The correction for 1000 grm. of substance weighed in dry air at 20° and 760 m.m. is stated for each substance.

Substance.	Density at 20°.	Correction for 1 grm. of substance, 20° 760 m.m.
Silver	10.49	- 0.000029
Sodic chloride ..	2.14	+ 0.0000418
Silver chloride ..	5.56(a)	+ 0.000073
Weights	8.40	—

(a) Richards and Stull, in an investigation as yet unpublished.

Especially when sodic chloride or silver chloride was weighed the temperature and barometric pressure were recorded at the time of weighing. In case of significant variations of temperature or pressure the corrections were changed accordingly. The correction is computed as follows:—

Correction = w (volume of 1 grm. of substance - volume 1 grm. of weights), where w is the weight of 1 c.c. of air at the given temperature and pressure. The following small table contains a sufficiently accurate statement of this value in convenient form for use with a logarithmic slide rule.

Value of w in Grms.			
Temperature (°C.).	750 m.m.	760 m.m.	670 m.m.
14°	0.001214	0.001230	0.001246
16°	0.001205	0.001221	0.001237
18°	0.001197	0.001213	0.001229
20°	0.001189	0.001204	0.001220
22°	0.001181	0.001196	0.001212
24°	0.001173	0.001188	0.001204

If the air is completely saturated with water vapour the above values should be decreased by 0.000008 at 14°, 0.000010 at 20°, and 0.000013 at 24°. But since the air of our balance cases was certainly less than a quarter saturated with moisture, any correction on this account would have been supererogatory.

(To be continued).

NOTICES OF BOOKS.

Practical Exercises in Chemistry. By G. C. DONINGTON, M.A. London: Macmillan and Co., Ltd. New York: The Macmillan Company. 1906.

THIS book will be found a most valuable aid to the teacher of large classes, for it is one that may safely be put into the hands of the pupil, and may be trusted to give him sufficient—but not superfluous—instructions for his experimental work, and to develop in him the spirit of research. It is undoubtedly one of the best of its kind that we have come across, and can be thoroughly recommended, especially for the use of older students. The subject-matter includes all that is required for the examination in chemistry of the London University Matriculation. The first four chapters are introductory, dealing with the measurement of volume, capacity and density, and simple chemical operations; then the properties and behaviour of common materials, rocks and earths, salts, acids, &c., are taken up, followed by the study of rusting and burning; this order possesses many advantages over the older plan of performing experiments on oxidation at the very beginning of the study of practical chemistry; the learner's interest in the subject is thoroughly awakened, and when he approaches the more difficult work he is much better equipped to profit by it.

Third Treatise on the Effects of Borax and Boracic Acid on the Human System. By Dr. OSCAR LIEBREICH. Translated from the German. London: J. and A. Churchill. 1906.

THE object of this treatise is to review and criticise Dr. Wiley's report, which was issued in 1904, on the influence of boron compounds on health and digestion. Dr. Liebreich does not agree with the conclusions drawn from

the Washington experiments, and in many respects it must be confessed that he is justified in the attitude he adopts towards the results of the experiments. For instance, he regards the observations as not sufficiently extended, and lays emphasis on the fact that the method employed was purely chemical, while clinical and medical reports, which would have been of immense value, were not prepared. He criticises, also, the form of administration of the boron compounds, which certainly left something to be desired, and he does not consider that the hygienic arrangements were by any means satisfactory. There can be no doubt that a therapeutic test would have furnished more reliable data, and Dr. Liebreich appears to state his case with moderation, and to have estimated correctly the true value of the experiments and Dr. Wiley's report on them.

Foods and Food Control. U.S. Department of Agriculture, Bureau of Chemistry, Bulletin No. 69. Parts I. to VIII. By W. D. BIGELOW. Washington: Government Printing Office. 1905.

THIS compilation of the laws relating to food products, arranged under the headings of the various States, will be found very valuable by manufacturers and dealers who now experience some difficulty in keeping themselves informed of the exact details of the law as regards these substances, on account of the lack of uniformity in the different States and the constant alterations of the regulations. Consumers may also find the compilation useful, and it will be much appreciated by food inspectors. The first edition of the Bulletin was issued in 1902, since which date many legislative changes have been made, so much so that Dr. Wiley, the Chief of the Bureau of Chemistry, considered it advisable to revise the entire Bulletin, instead of merely printing the new laws and amendments, so that these five volumes give a complete review of all the laws now in force in each State, besides the Federal laws.

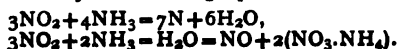
CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

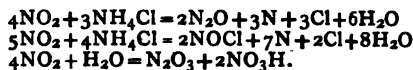
Comptes Rendus Hebdomadaires des Séances de l'Académie des Sciences. Vol. cxlii., No. 11, March 12, 1906.

Action of Hot Sulphuric Acid on Platinum and Iridium Salts in presence of Ammonium Sulphate.—Marcel Delépine.—The author isolates three species of irido-sulphuric salts, all strongly coloured. 1. Greenish blue salts which give a blue precipitate with salts of barium and strontium, and are decomposed by cold ammonia solution, giving a violet colouration. 2. Green salts which are not precipitated by neutral barium chloride, but can be precipitated to a greenish brown colour in an alkaline medium. The precipitate re-dissolves in acids, the green colour returning. These green salts also give with ammonia an olive-brown solution, which in time becomes greener, and is not precipitated by barium chloride, even when alkaline. 3. Olive brown salts which are not precipitated by neutral barium chloride, but give a brown precipitate in presence of ammonia. The solution darkens a little on the addition of alkalis. In all these bodies the sulphuric acid is apparently masked.

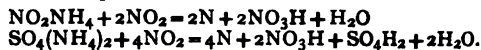
Action of Nitrogen Peroxide on Ammonia and certain Ammoniacal Salts.—MM. Besson and Rossel.—The principal reactions of ammonia gas on nitrogen peroxide under the conditions described by the author can be represented by the following equations:—



When the peroxide acts on ammonium chloride, the reactions are as follows:—



The peroxide of nitrogen acts on the nitrate and sulphate of ammonia under the same conditions as the chloride, but the gas evolved is exclusively nitrogen. The liquefied mass forms two superposed layers, of which the upper is formed of excess of peroxide; after elimination by distillation, in the first case nitric acid remains, and a mixture of nitric and sulphuric in the second:—



Action of Silicon Chloride on Cobalt.—Em. Vigouroux.—At a high temperature silicon chloride is reduced by cobalt, with formation of metallic chloride (which volatilises) and cobalt silicon (which remains). At about 1200–1300° the action ceases—that is to say, when the saturation is completed, and when the alloy contains about 19 to 20 per cent of combined silicon. This limit of siliciuration corresponds to the formula Co_2Si .

Dilactide of Lævo-lactic Acid.—E. Jungfleisch and M. Godchot.—In a previous research the authors described the preparation of the dilactide formed from dextro-lactic acid. They now prepare the corresponding compound produced from lævo-lactic acid. After purifications by crystallisation from ether, the *l*-dilactide forms crystals similar in appearance to those of the *d*-dilactide. The two dilactides combine to form (*d* + *l*) dilactide. This is much less soluble in ether than the active components. Combination easily takes place by separately dissolving equal quantities of the components in cold ether and mixing the two. Crystals of (*d* + *l*) dilactide are deposited.

MEETINGS FOR THE WEEK.

WEDNESDAY, 18th.—Microscopical, 8. Exhibition of Lantern Slides of Plant Structure, prepared by A. Flinters.

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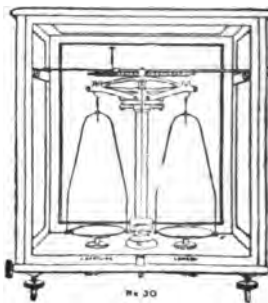
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THE CHEMICAL NEWS.

VOL. XCIII., No. 2421.

A SYSTEM OF QUALITATIVE ANALYSIS INCLUDING NEARLY ALL THE METALLIC ELEMENTS.*

PART II.—ANALYSIS OF THE TUNGSTEN GROUP.

By ARTHUR A. NOYES.

Introduction.

THE work upon the separation and detection of the elements whose hydroxides after heating at 120° are undissolved by nitric acid, constituting what for brevity is here designated the tungsten group,† has now been completed; but the method of procedure is still subject to revision as a result of information that may be gained in working out more fully the later groups, and additional test analyses covering other combinations of elements will be made as opportunity occurs. This publication is therefore to a certain extent a preliminary one.

The plan of presentation adopted in Part I. has been adhered to, special chapters being devoted to a "Tabular Outline" of the process, to a "General Discussion" of its main features, to the detailed "Procedure and Notes," to "Test Analyses" showing the efficiency of the method, and to "Confirmatory Experiments and References," substantiating the statements made in the notes.

I desire to state that during the first part of the work connected with this group Mr. B. E. Schlesinger was associated with me, and that to him are due the working out of the main sub-divisions of the group, and the discovery of the method of detecting tantalum in the presence of titanium. In the latter part of the work, consisting in perfecting the scheme and especially in developing the means of detecting niobium, tungsten, and tin, I have been ably assisted by Mr. D. P. Smith, and Mr. C. S. Bryan, jun. I also wish to express my thanks to Mr. F. M. Eaton for the valuable aid he has rendered by making a large number of test analyses by the final procedure.

Tabular Outline.

An outline of the process finally adopted is given in the table. Enclosure in brackets shows that the element may divide itself between the precipitate and filtrate at the point indicated.

General Discussion.

1. The residue insoluble in dilute nitric acid ordinarily contains all the tin, tungsten, tantalum, and niobium present in the substance, almost all of the antimony and titanium when these are present in even rather small quantities, much of the molybdenum and germanium when the substance contains a large quantity of them, and a greater or less proportion of many other elements, such as phosphorus, vanadium, arsenic, bismuth, selenium, tellurium, copper, iron, zirconium, which are carried down with the first-named elements. (See P. 5, N. 1, 3, 4, 5). Aside from the complications introduced by the occasional presence of these foreign elements, the difficulties involved in the separation, even sufficiently for qualitative purposes, of some of the regular elements of the group—especially of titanium, niobium, tantalum, and tungsten—have proved to be far greater than that of any other elements excepting the rare earths. And in spite of the existence of many analyses purporting to show the percentage composition of minerals containing two or more of these elements, it seems very doubtful whether any analytical method of separation has yet been developed by which quantitative results accurate even with in several per cent can be obtained. The

* From the *Technology Quarterly*, xvii., No. 3.
† Called the "niobium and tungsten groups" in Part I.

Tabular Outline.

Residue (SiO ₂ , TiO ₂ , Ta ₂ O ₅ , Nb ₂ O ₅ , Sb ₂ O ₃ , [Bi ₂ O ₃], SnO ₂ , [As ₂ O ₅], [P ₂ O ₅], [WO ₃], [MoO ₃], &c.).—Dissolve in HF, pass in H ₂ S, and filter. (P. 31).	Filtrate. (NH ₄) ₂ SnS ₃ , (NH ₄) ₂ SbS ₄ , [(NH ₄) ₂ WS ₄], [(NH ₄) ₂ MoS ₄], (NH ₄) ₃ AsS ₄ .
Precipitate ([Sb ₂ S ₃], [MoS ₃], [As ₂ S ₃], [Bi ₂ S ₃], &c.).—Boil with NH ₄ OH and filter. (P. 32).	
Residue. [MoO ₃], [H ₂ MoO ₄], [H ₃ SiO ₄], Bi(NO ₃) ₃ , H ₂ AsO ₄ . (P. 51).	
Precipitate. 2K ₂ TaF ₇ , Ta ₂ O ₅ . TA TEST.	
Residue (TiO ₂ , Nb ₂ O ₅)—Dissolve in HF, evaporate with H ₂ SO ₄ , dilute, pour through a Zn column, add HgCl ₂ . (P. 38).	Solution. —[Na ₂ WS ₄], [Na ₂ MoS ₄].
Precipitate.	Unite, acidify with H ₂ SO ₄ , shake, and filter. (P. 39).
Residue (TiO ₂ , Nb ₂ O ₅).	Precipitate (SnS ₂ , [WS ₃], MoS ₃ , As ₂ S ₃ , Sb ₂ S ₃).—Ignite in a current of H ₂ S gas. (P. 41).
Residue (TiO ₂ , Nb ₂ O ₅).	Sublimable. Residue (SnS, Sb ₂ S ₃ , WS ₂ , MoS ₂).—Boil with strong HCl and filter. (P. 41).
Residue (TiO ₂ , Nb ₂ O ₅).	Solution ([SnCl ₂], SbCl ₃).—Add HgCl ₂ . (P. 42).
Residue (TiO ₂ , Nb ₂ O ₅).	Precipitate —HgCl. SN TEST.
Residue (TiO ₂ , Nb ₂ O ₅).	Residue (WS ₂ , MoS ₂).—Heat with HNO ₃ and H ₂ SO ₄ . (P. 43).
Residue (TiO ₂ , Nb ₂ O ₅).	Solution (H ₂ MoO ₄).—Add Zn and KSCN. (P. 45).
Residue (TiO ₂ , Nb ₂ O ₅).	Mo TEST.
Residue (TiO ₂ , Nb ₂ O ₅).	Residue (H ₂ WO ₄). W TEST.
Residue (TiO ₂ , Nb ₂ O ₅).	Precipitate (H ₂ WO ₄). W TEST.
Residue (TiO ₂ , Nb ₂ O ₅).	Add HCl and Zn. (P. 44).
Residue (TiO ₂ , Nb ₂ O ₅).	W CONFIRMATORY TEST.

properties of these elements are therefore well worthy of more thorough going investigation from this point of view. The reader must not therefore be surprised to find that the scheme here proposed for the qualitative analysis of this group is somewhat complicated, and that it is not always possible to detect by it quantities as small as 1 or 2 m.grms. of some of these elements, when a large quantity of some other element is present.

2. The elements of this group are at the start present in hydrofluoric acid solution, the oxides having been treated with this solvent (P. 5) to remove silica, and to separate them from any undecomposed portion of the original substance. As experiments showed that the regular elements of the group, except antimony and molybdenum, are entirely unprecipitated by hydrogen sulphide from such a solution, while some of the foreign elements, bismuth, copper, selenium, and tellurium, are completely thrown out by it, it is thought best to submit the solution first to the action of this reagent. By this operation any complications that might arise in the later procedures from the presence of these elements are at once eliminated. A large part of the arsenic, antimony, and molybdenum is also removed, and this may be added to the main nitric acid solution, thereby making it unnecessary to test for these elements later in this group. Moreover, this treatment prevents so much molybdenum from being retained with the titanium in the ammonium sulphide precipitate, which otherwise is a rather serious difficulty when a very large quantity of this element is present. It is probable that this method, involving the use of hydrogen sulphide in hydrofluoric acid solution can be advantageously employed also for the quantitative separation of tin and tungsten from many of the other elements ordinarily precipitated by that reagent; it is not, however, possible in this way to separate those elements from antimony, arsenic, or molybdenum, since these are not by any means completely precipitated in the presence of hydrofluoric acid. The investigation of such quantitative methods can not be included within the scope of this research; but reference will occasionally be made to those which seem promising, in the hope that their study may be undertaken by others.

3. The solution is next evaporated with sulphuric acid to remove the hydrofluoric acid and decompose any fluosilicic acid. Sulphuric acid is used rather than nitric or hydrochloric, since it holds in solution, even after dilution, moderate quantities of all the elements of these groups except tungsten. Experiments were made in the hope of accomplishing the separation and detection of tungsten at this point; but, although when present alone 2 m.grms. give rise to a precipitate when the diluted sulphuric acid solution is allowed to stand, yet in the presence of titanium a much larger quantity remains in solution.

4. The method that naturally suggests itself for the subdivision of this group is digestion with ammonium sulphide solution, or fusion with sodium carbonate and sulphur followed by treatment with water, whereby titanium, niobium, and tantalum might be expected to remain undissolved, and tin, antimony, arsenic, tungsten, and molybdenum to pass into solution. This method is, in fact, often employed for the separation of titanium from tin and tungsten. Our experiments, like those of some previous investigators, have shown, however, that a considerable quantity of tungsten is retained in the titanium hydroxide precipitate, and that even 5 m.grms. of it may be removed so completely from the sulphide solution that it cannot be detected in the filtrate. Nevertheless, we have found no better method of separation than this, and have therefore made it an important part of the scheme of analysis for this group. This is further justified by the fact that the presence of tungsten in the titanium precipitate is indicated in the subsequent procedures, and that then it can, to a large extent, be separated from the titanium by fusion with sodium carbonate and sulphur, and be subsequently detected. The separation of tin from titanium by ammonium sulphide seems to be more satisfactory. Experiments on the method used by Defacqz for the separation

of titanium and tungsten, consisting in fusion of the oxides with a mixture of potassium nitrate and carbonate, did not give so satisfactory results.

5. The quantitative separation in the wet way of the three elements, titanium, niobium, and tantalum, present in the ammonium sulphide precipitate, is also, we believe, still an unsolved problem, and even the qualitative detection of small quantities of niobium and tantalum in the presence of much titanium presented so many difficulties that it was for a long time feared that the attempt to accomplish it would have to be abandoned. The method usually employed for the separation of niobium from tantalum, based upon the different solubilities of the potassium fluoxyniobate and fluoxytantalate, gives satisfactory results; but experiments showed that this method of conversion into double fluorides was not available for the separation of either of these elements from a large quantity of titanium, owing to the intermediate solubility of the potassium fluotitanate. The common quantitative method of separating titanium from the other two elements, by fusing the oxides with potassium disulphate and extracting with cold water the titanium sulphate, while leaving tantalum and niobium oxides undissolved, was found to be useless, since fairly large quantities of these elements also pass into solution when much titanium is present. Failure also attended attempts to separate these elements by treating their dried hydroxides with acidified hydrogen peroxide, and also by dropping an acid peroxide solution of them into a strongly ammoniacal solution of hydrogen peroxide, according to the method used by Walker for the separation of iron and titanium.

6. It was fortunately discovered, however, that an acid solution of hydrogen peroxide dissolves even large quantities of the precipitated hydroxides of niobium and titanium readily, and nearly completely, at any rate upon a repetition of the precipitation and treatment with peroxide; but that tantalum hydroxide, whether present in small or large quantity, whether alone or with much titanium, remains in large part (about one-half) undissolved by this solvent. It can therefore be detected in the residue by conversion into the insoluble potassium fluoxytantalate. The dissolved part does not, moreover, interfere with the tests adopted for the other two elements.

7. It then remained to devise a plan for the detection of titanium and niobium. No satisfactory method of separation could be found. It was proved, indeed, when the hydrogen peroxide was reduced by sulphurous acid and the solution made alkaline by ammonium hydroxide, that the precipitated hydroxide of titanium dissolved completely on strongly acidifying the solution with hydrochloric acid, while that of niobium, when present alone, remained in large part undissolved. But when much titanium was present with the niobium, considerable quantities of the latter went completely into solution, so that the method was unsuitable.

8. The attempt to effect a separation of the two elements was then given up, and characteristic reactions which would show one in the presence of the other were sought for. The yellow colour imparted to the peroxide solution is sufficiently characteristic of titanium, as neither tantalum nor niobium gives any colour to it. There seems to exist, however, no colour reaction given by niobium with a reagent which does not affect titanium. The idea then presented itself that the lower oxides, which are produced in the case of both elements by the action of zinc on acid solutions of the ordinary hydroxides, might possess markedly different reducing powers. This was found to be the case, for the lower oxide of titanium reduces mercuric chloride only very slowly, while that of niobium causes instantaneous reduction. Unfortunately, tungsten and molybdenum show the same behaviour as niobium, and they are apt to be retained in the ammonium sulphide precipitate; but it was found possible to remove them almost completely by the fusion with sodium carbonate and sulphur above referred to, so that error from this source may be avoided. It seems probable that the power of the

lower oxide of niobium of reducing mercuric chloride might be used for the quantitative estimation of this element in the presence of tantalum and titanium perhaps best by weighing the precipitated mercurous chloride.

9. Difficulty was also met with in the isolation of the tungsten from the alkaline sulphide solution; for upon acidification a large part, or if it is present in small quantity, almost the whole of that element, remains in solution, probably in a colloidal condition. Much more tungsten is precipitated if ferric chloride is added to the acidified solution, and the mixture is shaken and allowed to stand; but to detect small quantities of tungsten, it is even then necessary to recover it from the filtrate, so that the use of ferric chloride is not ordinarily to be recommended. The filtrate is merely evaporated, the residue ignited and fused with sodium carbonate, and the aqueous solution of it immediately tested for tungsten by acidification; so that no complications other than are involved in the performance of a few additional operations are introduced.

10. The sulphide precipitate may contain tin, antimony, tungsten, molybdenum, arsenic, and vanadium. It was found that the separation of the first two elements from any of the others (except molybdenum) by heating with concentrated hydrochloric acid was very imperfect, owing to the not inconsiderable solubility of the moist sulphides of tungsten, vanadium, and arsenic in that solvent. Fairly large quantities of tungsten especially went into solution when accompanied by a large quantity of tin, and thus escaped detection. On the other hand, the process sometimes used for the quantitative separation of tin and tungsten, which consists in igniting the sulphides in a current of hydrogen sulphide, and then treating them with strong hydrochloric acid, was found to give excellent results with all the metals concerned, and was adopted as part of the procedure. Incidentally it has the advantage of volatilising any arsenic present, which might otherwise prevent the separation of even a large quantity of tungstic acid in the later treatment of the sulphides with nitric acid.

11. The only other matter which requires mention is the separation of tungsten and molybdenum. When the latter element is absent, or present only in small quantity, tungstic acid alone separates as a yellow powder, upon treating with nitric acid the sulphides from which the antimony and tin have been previously extracted with hydrochloric acid; but if much molybdenum is present, even a considerable quantity of tungsten remains in solution, and if the solution is evaporated the molybdenum itself separates, after which it re-dissolves in acid with great difficulty. Some supplementary method of separation is therefore necessary. The most suitable one found is that of Ruegenberg and Smith as modified by Hommel, which consists merely in evaporating with sulphuric acid nearly to the fuming point, digesting for a time, and then diluting, whereby the tungsten is precipitated and the molybdenum retained in solution. The separation is not very sharp; yet as little as 3 m.grms. of tungsten always precipitate, while even a large quantity of molybdenum does not do so.

(To be continued).

Royal Institution.—On Tuesday next, April 24, at 5 o'clock, Prof. G. Baldwin Brown will commence a course of two lectures at the Royal Institution on "Greek Classical Dress in Life and in Art"; on Thursday, April 26, at the same hour, Dr. P. Chalmers Mitchell will deliver the first of two lectures on "The Digestive Tract in Birds and Mammals"; and on Saturday, April 28, at 3 o'clock, Prof. Charles Waldstein begins a course of three lectures on "English Furniture in the Eighteenth Century." The Friday Evening Discourses will be resumed on April 27, when Prof. J. W. Gregory will deliver a discourse on "Ore Deposits and their Distribution in Depth." The discourse on May 4 will be given by the Hon. C. A. Parsons on "The Steam Turbine on Land and at Sea," and on May 11 by Prof. J. P. Poynting on "Some Astronomical Consequences of the Pressure of Light."

A REVISION OF THE ATOMIC WEIGHTS OF SODIUM AND CHLORINE.*

By THEODORE WILLIAM RICHARDS
and
ROGER CLARK WELLS.

(Continued from p. 177).

PREPARATION OF PURE MATERIALS.

Water.

THE traces of organic impurity in the distilled water of the laboratory were eliminated by re-distilling from a weakly alkaline solution of potassic permanganate, using at first a glass condenser, and rejecting the first quarter of the distillate. Water thus prepared was pure enough for preliminary nephelometric work, when the presence of a trace of alkali could do no harm; it was usually kept in Jena flasks. In cases where the greatest purity was desired it was distilled, of course, once further, and for the nephelometric experiments it was kept in a closed bottle provided with a syphon and protected from possible traces of hydrochloric acid by a tube containing sodic hydroxide. For final preparations and accurate analyses, where silica and alkali must be excluded, the water was condensed and collected during the third distillation wholly in platinum. Care was taken to exclude dust in all stages of the proceedings; for, although visible dust might weigh little, its presence in silver halogen salt would produce decomposition and loss of weight in the final fusion. Only one who has attempted to exclude all dust from solutions can appreciate the difficulty of doing so. Thorpe and Roger (*Phil. Trans.*, 1894, A, clxxxv., 432), working upon viscosities, found that sealed-in condensers were very helpful in excluding dust from their distillates. We did not go as far as this, but the danger was borne in mind at every stage of the work and guarded against.

Hydrochloric Acid.

For the final work the purest acid of commerce was treated with a few crystals of potassic permanganate, diluted, and boiled. This should have expelled bromine and iodine, and must have oxidised any trace of organic matter which might have been present. The oxidation and boiling were repeated; the solution was fractionally distilled; and for the succeeding treatment the middle fraction was selected and subjected to another distillation, in which a platinum condenser and receiver were employed. As a matter of fact, most of these precautions were found to be unnecessary, since salt made, as described under "Sodic Chloride," from the best "chemically" pure acid of commerce only once re-distilled in platinum, without any further precautions, gave the same results as the purest material.

Sodic Chloride.

In most cases a perfectly pure salt cannot be made by precipitating the impurities, as many are prone to assume, but must itself be precipitated or crystallised from the less pure solution. Sodic chloride is nearly insoluble in a concentrated solution of hydrochloric acid; therefore the usual method of precipitation by means of hydrochloric gas seemed the best possible method of obtaining pure sodic chloride, since any included hydrochloric acid could be completely expelled by fusion. This expectation was confirmed by trial, and when it had been found that this means of purification was unusually suitable, most of the specimens of salt were treated in this way. This and other purifications were greatly assisted by centrifugal draining of the crystals. For this purpose salt was collected in a platinum funnel, and this was covered and supported on a stout frame capable of being whirled by means of strong cord about a radius of a metre. A test-tube, firmly fixed in the frame, collected the separated mother-liquor. Centrifugal draining, so important in technical work, has hardly received the attention which it

* Published by the Carnegie Institution of Washington, April, 1905.

deserves in the scientific laboratory. Experiments have shown that a good centrifugal apparatus is capable of separating nine-tenths or more, according to the habit of the crystals, of the mother-liquor which would otherwise contaminate the solid; hence making the purification at least ten times as effective as it would otherwise be (Richards, *Journ. Am. Chem. Soc.*, 1905, xxvii., 104). The whole treatment of precipitation and draining was carried on in platinum vessels which had been effectively freed from superficial iron.

Besides this chief mode of purification, others were used in special cases, and the original material came from a variety of sources, as recorded below. One of these was Merck's purest sodic chloride, which this firm stated had been prepared from German rock salt. Another was the very pure sodic sulphate prepared for the experiments on the transition temperature of this substance, and known to be very pure, because of the constancy and accuracy of its transition point (32.383°) (Richards and Wells, *Proc. Am. Acad.*, 1902, xxxviii., 431; also *Zeit. Phys. Chem.*, 1903, xliii., 465). Another was a fine specimen of Stassfurt halite, perfectly clear and colourless. A fourth specimen was made from the purest sodic bicarbonate manufactured by the Solvay Process Company, of Syracuse, New York, and very kindly furnished in large quantity by that company. A fifth sample was made from pure sodic carbonate from Merck. These preparations, differing widely in the steps of manufacture, and in geographical source, all yielded essentially the same atomic weight.

The source of the materials having been given above, it remains to record the details of their preparation.

In the first place, a large quantity of Merck's sodic chloride, which had been purified from German rock salt, was thrice re-precipitated by hydrochloric acid. This salt is designated "sample A" below, and was used in preliminary experiments. It was further crystallised from water (B); some of this was fused with ammoniac chloride and ammoniac chlorplatinate (C); a portion was then precipitated with hydrochloric acid (D); and finally some was again fractionated from water (E). None of these operations seemed to have any essential effect upon the combining weight of the salt; hence, even sample A must have been practically pure.

The very pure sodic sulphate was easily converted into chloride by successive precipitation from solution by means of gaseous hydrochloric acid. The sulphate forms an especially fortunate source of pure sodium, since none of the other alkaline sulphates yield crystals of the same degree of hydration or the same crystal form. Hence sodic sulphate which has been many times re-crystallised must be extremely pure. The ease with which a constant transition temperature may be obtained is evidence of this.

A specimen of sodic sulphate, which had been shown to be pure by its transition temperature, was twice further re-crystallised before adding hydrochloric acid. Four precipitations by this gas, with washings and drainings, removed practically all of the sulphuric acid. Nevertheless, when the mother-liquor was concentrated by evaporation and tested, an extremely small trace of sulphate was found by excess of baric salt to persist until the tenth precipitation. There was no trace of visible baric sulphate on testing the mother-liquor, even in the nephelometer, after the eleventh precipitation, but the salt was precipitated once more for certainty. This salt, thus twelve times separated by hydrochloric acid, was designated F. It seems highly probable that since so large an amount of sulphuric acid was eliminated from it by this treatment, all other impurities must have been separated.

For another specimen of salt, a number of fine, large, colourless transparent crystals of halite, from near Stassfurt, Germany, were dissolved in pure water. After the addition of a little sodic hydroxide, boiling, and standing, the solution was filtered and precipitated with hydrochloric gas. A solution of the latter salt in water stood several days. The clear decanted portion was precipitated twice more, fractionally crystallised from

water, and dried over sodic hydroxide in a vacuum desiccator. This specimen was called sample G.

The bicarbonate from Syracuse, New York, was of the following composition, according to the report of the Solvay Process Company:—

	Percent.
Silica	0.006
Ferric oxide and alumina	0.002
Calcic carbonate	0.030
Magnesian carbonate	0.005
Sodic chloride	0.070
Sodic carbonate	0.522
Sodic bicarbonate	99.300

This material, already very pure for a commercial substance, was easily further purified by repeated washing with cold water, which was sufficient to remove most of the chloride.

A portion of the Syracuse bicarbonate, thus washed four times with cold water and thoroughly drained each time with a reverse platinum filter, was nearly all dissolved in water. After settling, the resulting clear solution was decanted, neutralised with hydrochloric acid, and then evaporated to dryness. The sodic chloride was fused in platinum, dissolved, and considerably diluted. After long standing the clear upper portion was decanted from very finely divided silica. This method of fusion is a very effective means of separating silica, but the precipitate is so fine as to require a long time to settle, and has to run through most filtering septa. Hence, cautious decanting, with rejection of the considerable lower portion of the solution, is necessary. The unimpeachable solution of salt thus obtained was concentrated and twice precipitated by hydrochloric gas (H), twice more precipitated (I), and yet twice more precipitated (J). Analysis showed these three to be alike, as will be recorded.

The Merck bicarbonate was thoroughly washed and once re-crystallised from water. It was then gently ignited, and the normal carbonate was three times crystallised from water. From this carbonate, by the usual process of precipitation by hydrochloric gas and crystallisation from water, sample K was prepared. This sample gave essentially the same combining number as the others.

With such conclusive evidence that no foreign base was present, we turned our attention to the acid. The preceding preparations had all been precipitated with hydrochloric acid gas, obtained simply by warming the highly concentrated purest acid of commerce, because we deemed it more convenient to use this constant material, even if slightly impure, than to prepare so large a quantity of the purest acid. A special set of comparative experiments, made with the purest acid, proved that the other material had, as a matter of fact, been pure enough for the most exacting requirements.

For this purpose the very pure hydrochloric acid was used whose preparation is described above. This acid was neutralised by an especially prepared sample of soda, made from a second portion of the Syracuse bicarbonate. The purification was conducted wholly in platinum vessels, of course. After being washed as before, the bicarbonate was dried and heated, to convert it into the normal carbonate. The solution of the latter in water was decanted daily as long as long as any precipitated residue (calcic carbonate) appeared visible. The sodic carbonate was then re-crystallised four times, as the deka- or hepta-hydrate, and once as the anhydrous salt. A concentrated solution of the latter salt was then treated for a considerable time with carbon dioxide under a bell-jar until the bicarbonate had formed. The carbon dioxide was made by gently heating sodic bicarbonate, and was well washed with pure water. The pure bicarbonate thus formed was washed with cold water, thoroughly drained, and dissolved in an excess of the pure hydrochloric acid. The resulting salt was twice fractionally re-crystallised from water on the steam bath; and the crystals were washed and drained each time, in order to free them from any possible calcium or magnesium chlorides, which are not easily eliminated from carbonate.

Since the atomic weight from this preparation (L) was identical with that from the preceding preparations, the previous salts, made by precipitation with hydrochloric gas, could have contained no other halogen than chlorine.

It appears from the identity of all these specimens that sodic chloride is among the substances whose preparation in a pure state is an easy problem. It is further true that the expulsion of water from it upon fusion without loss of halogen is a very simple matter; moreover, since we succeeded in proving that salt fused in a vacuum possesses the same combining weight as that fused in air, the solid contains neither occluded oxygen nor nitrogen in weighable amounts. This matter is discussed later on.

Considering these experiments, it is fairly safe to infer that all the various preparations of sodic chloride were quite free from significant quantities of any foreign substance.

(To be continued).

THIRTEENTH ANNUAL REPORT OF THE COMMITTEE ON ATOMIC WEIGHTS. DETERMINATIONS PUBLISHED IN 1905.*

By F. W. CLARKE.

(Continued from p. 171).

Bromine.

In his research upon the atomic weight of iodine, Baxter (*Journ. Am. Chem. Soc.*, xxvii., 884) made a series of experiments upon the conversion of silver bromide into chloride by heating in a current of dry chlorine. Vacuum weights are given in the following table, and reduced with $Ag = 107.93$ and $Cl = 35.473$.

Weight AgBr.	Weight AgCl.	Atomic weight Br.
10.92091	8.33538	79.955
13.88062	10.59457	79.951
8.21484	6.27006	79.952
7.87887	6.01352	79.956
6.90106	5.26735	79.951
9.53704	7.27926	79.952
Mean		79.953

This is sensibly identical with the generally accepted value, 79.955.

Iodine.

An important continuation of the research upon iodine, which was reported in 1904, has been published by Baxter (*Journ. Am. Chem. Soc.*, xxvii., 876). First, silver iodide was converted into bromide by heating in bromine. The corrected data are as follows:—

Weight AgI.	Weight AgBr.	Atomic weight I.
13.65457	10.92091	126.985
17.35528	13.88062	126.987
9.70100	7.75896	126.982
10.27105	8.21484	126.983
9.85688	7.88351	126.986
8.62870	6.90106	126.991
11.92405	9.53704	126.981
7.56933	6.05389	126.987
Mean		126.985

Atomic weights calculated with $Ag = 107.93$, $Br = 79.955$.

Two new series of determinations were made of the ratio $AgI : AgCl$. The first of these was upon the weights of iodide given in the foregoing table; that is, the bromide, after weighing, was transformed into chloride.

* From the *Journal of the American Chemical Society*, xxviii., No. 3.

Weight AgI.	Weight AgCl.	Atomic weight I.
Series II.		
13.65457	8.33538	126.985
17.35528	10.59457	126.983
10.27105	6.27006	126.980
8.62870	5.26735	126.985
11.92405	7.27926	126.976
Mean		126.982
Series III.		
9.26860	5.65787	126.990
6.72061	4.10259	126.984
11.31825	6.90912	126.987
10.07029	6.14754	126.979
13.49229	8.23649	126.980
Mean		126.984

Three additional series of measurements relate to the ratio between silver and iodine. In the first one, iodine and silver were weighed separately; the iodine was then converted into hydriodic acid and precipitated by known quantities of silver.

Weight I.	Weight Ag.	Atomic weight I.
3.29308	2.79897	126.983
3.70132	3.14584	126.988
3.75641	3.19258	126.991
3.24954	2.76186	126.988
4.12541	3.50639	126.984
3.53166	3.00165	126.988
2.99835	2.54842	126.985
2.00015	1.69991	126.993
Mean		126.987

In five of these experiments the silver iodide was collected and weighed, with the subjoined results.

Weight I.	Weight AgI.	Atomic weight I.
3.75641	6.94917	126.987
3.24954	6.01137	126.989
4.12541	7.63204	126.977
3.53166	6.53351	126.979
2.99835	5.54682	126.983
Mean		126.983

Finally, in order to determine the ratio $Ag : AgI$, the filtrate and washings from the previous sets of analyses were examined for the silver they might contain. This gave a small correction to the weights already cited, with results as follows:—

Weight Ag.	Weight AgI.	Atomic weight I.
3.19249	6.94877	126.990
2.76175	6.01110	126.986
3.00189	6.53399	126.993
2.54833	5.54659	126.986
Mean		126.989

The final value adopted by Baxter is $I = 126.985$ when $Ag = 107.93$ and $Cl = 35.473$. Vacuum weights are given throughout the memoir. Two short critical papers on iodine, by Ladenburg (*Ann.*, cccxxxviii., 259) and Koethner (*Ann.*, cccxxxviii., 262) respectively, have also appeared during the year. They contain no new determinations.

Sodium.

The determinations of the atomic weight of sodium by Richards and Wells cover the measurement of two ratios (*Journ. Am. Chem. Soc.*, xxvii., 459; *CHEMICAL NEWS*, xciii., p. 175; original in Publication 28 of the Carnegie Institution). It goes without saying that in all of Richards' work every conceivable precaution is taken and every cor-

rection applied. The data obtained appear in the two following tables:—

Ratio NaCl : AgCl.		
Weight NaCl.	Weight AgCl.	Ratio, 100 AgCl : NaCl.
3·27527	8·03143	40·781
5·56875	13·65609	40·779
4·18052	10·25176	40·779
4·54319	11·14095	40·779
1·97447	4·84190	40·778
3·97442	9·74547	40·782
6·69495	16·41725	40·780
2·88692	7·07955	40·778
5·56991	13·65833	40·780
5·85900	14·36693	40·781
Mean		40·780

Ratio Ag : NaCl.		
Weight NaCl.	Weight Ag.	Ratio, 100 Ag : NaCl.
3·96051	7·30896	54·187
2·32651	4·29355	54·186
5·36802	9·90699	54·184
4·00548	7·39210	54·186
4·69304	8·66101	54·186
3·27189	6·03842	54·185
5·08685	9·38795	54·185
3·66793	6·76952	54·183
5·48890	10·12993	54·185
3·55943	6·56909	54·185
Mean		54·185

Two supplementary analyses for fixing the foregoing ratio were made with salt and silver which had both been fused *in vacuo*.

NaCl.	Ag.	Ratio.
3·38684	6·25046	54·185
4·68529	8·64634	54·188

With Ag = 107·92 and Cl = 35·470 we have for Na, from AgCl ratio, Na = 23·004; from Ag ratio, Na = 23·007. With Ag = 107·93 and Cl = 35·473, Na = 23·008.

Potassium.

Atomic weight re-determined by Archibald from analyses of the chloride (*Trans. Roy. Soc. Canada*, 1904, Section III., p. 47). Two ratios were measured. The data given below represent vacuum weights.

Ratio AgCl : KCl.			
Weight AgCl.	Weight KCl.	Ratio.	Atomic weight K.
4·25916	2·21586	52·028	39·142
3·83250	1·99379	52·023	39·138
5·57396	2·89977	52·025	39·139
9·10362	4·73606	52·026	39·140

Ratio Ag : KCl.			
Weight Ag.	Weight KCl.	Ratio.	Atomic weight K.
3·20598	2·21586	69·116	39·142
2·88479	1·99379	69·114	39·140
4·19557	2·89977	69·115	39·141
6·85280	4·73606	69·111	39·137

In mean K = 39·140 when Ag = 107·93 and Cl = 35·455. With Cl = 35·473, this becomes K = 39·122.

Carbon and Glucinum.

Parsons (*Journ. Am. Chem. Soc.*, xxvii., 1204) has re-discussed his analyses of the basic acetate and acetyl-acetate of glucinum, which were noticed in the report for 1904. Combining the two equations yielded by the two observed ratios, and solving algebraically, the following values were simultaneously determined:—

$$\text{Gl} = 9·112 \quad \text{C} = 12·007.$$

From the density and critical constants of CO₂, Guye (*Comptes Rendus*, cxli., 1241) finds C = 12·003; and from C₂H₂, C = 12·002. Jaquered and Perrot (*Comptes Rendus*, cxli., 1542), by comparing the densities of gases at 1067°, find the molecular weight of CO to be 28·009, and of CO₂ 43·992. Hence C = 12·009 and 11·992.

Guye and Pintza (*Comptes Rendus*, cxli., 51) have also determined anew the density of carbon dioxide. One litre at normal temperature and pressure weighs 1·97684, 1·97676, 1·97681 grms.

Lord Rayleigh (*Phil. Trans.*, cciv., 369), studying the compressibility of gases, has deduced several molecular weights from measurements of density at ordinary pressures and very small pressures. The subjoined data refer to carbon monoxide and dioxide when O₂ = 32.

	Atmospheric P.	Very small P.
Carbon monoxide	28·000	28·003
Carbon dioxide	44·268	44·014

From the figures in the second column, CO—O gives C = 12·003. CO₂—O₂ = 12·014, 2CO₂—CO = 11·992. From the density of hydrogen at very small pressures, H₂ = 2·0173.

(To be continued).

CONGRESS OF APPLIED CHEMISTRY AT ROME.

THE MORE IMPORTANT QUESTIONS TO BE DISCUSSED AT THE SIXTH INTERNATIONAL CONGRESS.

THE Sixth International Congress of Applied Chemistry will be held at Rome from April 26th to May 3rd, under the distinguished patronage of His Majesty the King of Italy. The character of the Congress will be veritably international, for it has been supported by, and received many communications from, the chemists of England, France, Germany, Austria, Hungary, Belgium, Switzerland, Spain, Holland, Norway, Russia, Roumania, Greece, Tunis, Egypt, India, Cape Town, United States of America, Argentina, Brazil, Chili, and Uruguay, besides those of Italy.

The English Committee, which is composed of delegates of fifteen of the principal societies of chemistry and the allied sciences, has for its general hon. secretary C. G. Cresswell (Palace Chambers, Westminster, S.W.). We hear that a large party of English chemists will leave London on April 23rd, and, going by way of the Lake of Como, will join the Congress at Rome.

Among the more important communication which will be read before the Congress we may mention that of Sir William Ramsay, "Sur l'Epurage des Eaux d'Egoit"; that of Henri Moissan, of Paris, "Sur la Distillation des Metaux"; and that of A. Frank, of Berlin, "Sur l'Utilisation Directe de l'Azote de l'Atmosphère pour la Production des Engrais et de Produits Chimiques."

Among the more important communications from the English chemists we note the following:—"Acido Solforico Fermentazioni, by Dr. W. Stevens, of London; "The Systematic Study of Absorption Spectra as Applied to Determining Problems of Chemical Constitution in Colourless and Coloured Substances; "The Application of Photography to the Solution of Problems in Chemistry," by Prof. W. N. Hartley, of Dublin; "The Fight between Cane- and Beet-sugar," by Dr. S. Stein, of Liverpool; "The Most Recent Methods for the Electrolytic Refining of Copper," by Dr. S. Cowper Coles, of London; "On certain Cases of Hydrolysis," by Dr. V. H. Veley, of Oxford.

Also the following important communications from the American chemists:—"Fused Sodium Peroxide and its Use in Air Purification," by Prof. P. Herbert and R. Forreger, of New York; "Comparison of the Characters of Petroleum of Recent Development with that of the Older Sources of Supply," by Dr. R. Clifford, of New York;

"The Effect of Environment upon the Composition of Sugar-producing Plants; "Important Problems of Agricultural Chemistry in the United States"; "The Inspection of Food Products Imported into the United States"; "The Use of Sulphur Fumes in the Preparation of Food Products," by Dr. H. W. Wiley, of Washington; "Dry Lead Defecation in Optical Sugar Analysis," by Dr. W. Horne, of New York.

The chemists of India announce the following papers:—"A Comparative Study of the Chief Methods for Estimating the Quantity of the Various By-products of Spirituous Liquors, with Analyses"; "The Chemistry and Comparative Toxicity of the By-products (a) of Potable Spirits made in India, (b) of Spirits Imported into India from Europe," by Prof. C. H. Bedford, of Calcutta; "Analyses of Effluents of Septic Tanks for Sewage Disposal in India," by Dr. Leather, of Bombay; "The Toxic Principle in the Bitter Variety of Luffa *Aegyptiaca*," by Dr. C. L. Bose, of Calcutta.

We may also call attention to the following:—"Which are the Most Suitable Means for Retarding the Fermentation of Must in Hot Climates to Secure Wines of better Bouquet," by Prof. P. D. Hahn, of Cape Town; "On the Chemistry of the Lakes Employed in Dyeing," by Prof. Z. Pocopios, of Athens; "The Chemical Selection of the Cane," by Dr. Kobus, of Pekalongan (Java).

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Annual General Meeting, March 30th, 1906.

Professor R. MELDOLA, F.R.S., President, in the Chair.

PROF. J. J. SUDBOROUGH and Mr. J. L. BAKER were appointed Scrutators, and the Ballot was opened for the election of Officers and Council for the ensuing year.

The PRESIDENT presented the Report of the Council on the progress of the Society during the past twelve months. The adoption of the Report of the Council was proposed by Mr. R. J. FRISWELL, seconded by Dr. H. BRERETON BAKER, and carried unanimously.

Report of the Council.

The Council have the satisfaction of being able to report that the past year has been a prosperous one for the Society, the activity of which, as measured by the number of papers read and the number of Fellows on the list, has exceeded that of any previous year.

On the 31st December, 1904, the number of Fellows was 2711. During 1905, 164 Fellows were elected and 2 re-instated, thus making a gross total of 2877. The Society has lost 25 Fellows by death, 28 have resigned, the election of 3 has become void, 1 has withdrawn, and 35 have been removed for non-payment of Annual Subscriptions. The total number of Fellows, therefore, on the 31st December, 1905, was 2785, showing an increase of 74 over the number for the previous year.

The names of the deceased Fellows, with the dates of their election, are:—W. Ackroyd (1897), C. M. Blades (1885), J. F. Braga (1881), G. B. Buckton (1852), J. L. Bullock (1842), C. F. Burnard (1849), J. H. Calvert (1871), H. S. Carpenter (1875), J. Duncan (1863), H. S. Elworthy (1886), J. Epps (1885), E. Graham (1895), W. H. Greenwood (1873), F. W. Harrold (1891), F. M. Mercer (1884), M. Prasad (1903), A. B. Prescott (1876), W. T. Rickard (1845), R. Rose (1886), F. Shapley (1893), C. W. Sutton (1884), C. R. C. Tichborne (1863), L. White (1862), W. W. Will (1885), R. Yates (1874).

The small number of Fellows still living who were elected in the early days of the Society has been further reduced by the death of Mr. G. B. Buckton, who was elected in March, 1852, and of Mr. J. L. Bullock, elected in 1842. Since the close of 1905, the Society has had to

lament the loss of Professor Hermann Johann Philipp Sprengel, who was elected in December, 1864.

The number of Honorary and Foreign Members at the date of the last Annual General Meeting was 35. No names have been added to the list since then, but the Society has sustained a loss in the death of Professor P. T. Cleve, who was elected in February, 1883, and who died on June 18th, 1905. The work of the deceased Honorary and Foreign Member is to be commemorated by a Memorial Lecture, the delivery of which has been undertaken by Professor T. E. Thorpe.

During the year 1905, 233 scientific communications have been made to the Society, 191 of which have been published already in the *Transactions*, and abstracts of all have appeared in the *Proceedings*.

The volume of *Transactions* for 1905 contains 1936 pages, of which 1818 are occupied by 184 memoirs, the remaining 118 pages being devoted to the Wislizenus Memorial Lecture, the Obituary Notices, the Report of the Annual General Meeting, and the Presidential Address; the volume for the preceding year contains 175 memoirs, which occupy 1715 pages.

The *Journal* for 1905 contains also 4356 abstracts of papers published mainly in foreign journals, which extend to 1828 pages, whilst the abstracts for 1904 numbered 4617 and occupied 1920 pages.

The abstracts for 1905 may be classified as follows:—

PART I.		Pages.	No. of Abstracts.
Organic Chemistry		956	1727
PART II.			
General and Physical Chemistry ..			619
Inorganic Chemistry			562
Mineralogical Chemistry			71
Physiological Chemistry			467
Chemistry of Vegetable Physiology and Agriculture			295
Analytical Chemistry			615
		872	2629
Total in Parts I. and II. ..	1828		4356

In making comparison with the preceding year, it must be borne in mind that the 1904 volume contained the abstracts for thirteen months. Owing to the change in date of publication, no abstracts appeared between December 1st, 1903, and January 31st, 1904, so that the number of the *Journal* published on the latter date contained practically the abstracts of two months (see *Trans.*, 1905, lxxxvii., 539).

The Council regrets to announce that Dr. G. T. Morgan has found it necessary to resign the post of Editor of the Society's publications which he has occupied so creditably since the beginning of 1903. Although his resignation was received in September, 1905, Dr. Morgan kindly acted as Editor until the appointment of his successor, Dr. J. C. Cain.

The second part (Subject index) of the Collective Index for the decade 1893-1902 was issued in December, 1905, to those Fellows who had made application for it in accordance with the printed notices circulated with the monthly parts of the *Journal* subsequently to July, 1903, and the Council have pleasure in expressing the high appreciation of the ceaseless energy displayed by the Indexer, Mrs. Margaret Dougal, on the completion of this valuable work.

During the vacation of 1905, advantage was taken of the Doctorial Jubilee of Professor Adolph von Baeyer, and of the Professorial Jubilee of Professor Mendeléeff, to address letters of congratulation to these two distinguished Honorary and Foreign Members.

In June, 1905, the Council decided that the Ordinary Meetings of the Society should be held during the ensuing Session on the first and third Thursdays of the month at 8.30 p.m. The experiment of holding the meetings on

Wednesday afternoons at 5.30 p.m., alternately with Thursday evenings at 8 p.m., had been in operation since January, 1902, and from the fact that the average attendance of Fellows at the afternoon meetings had undergone steady diminution during this period, whilst the number of Fellows attending the evening meetings had increased, it has been concluded that a majority of the Fellows who make a practice of attending the meetings find the evening more convenient than the afternoon.

The Library has received an important addition by the generosity of Sir Henry E. Roscoe, who has presented to the Society a collection of 136 alchemical and early chemical works of great interest and value.

An increase in the use of the Library is recorded, 1108 books being borrowed during 1905, as against 1057 in the previous year. Additions to the Library, excluding Sir Henry Roscoe's donation, comprise 165 books, of which 58 were presented, 324 volumes of periodicals, and 48 pamphlets, as against 119 books, 296 volumes of periodicals, and 52 pamphlets last year. On the recommendation of the Library Committee the Council have made an addition to the Library Rules in the following terms:—"No persons other than Fellows of the Society have the privilege of using the Library, except upon a written introduction from a Fellow, with whom rests the responsibility for all books consulted by the person introduced. Such introduction shall be valid for one occasion only."

Grants amounting in all to £236 have been made during the year from the Research Fund, and £22 15s. 3d. has been returned. Of the papers published in the *Transactions* during 1905, 24 were contributed by authors to whom grants had been made from the Research Fund.

In February of last year the Treasurer was fortunate enough to be able to increase the invested capital of the Society by almost exactly £1500 by the purchase of £1983 Midland Railway 2½ per cent Preference Stock for £1499 14s. 5d. The income from all sources for the year 1905 exceeds that for 1904 by only £93 8s. 4d., whilst the expenditure has been abnormally heavy, exceeding the total income by £305 15s. 5d. This is due to the incidence of several very heavy accounts, one of which really belongs to several years, and that is the completion of the Decennial Index for the period 1893-1902, which has entailed the expenditure this year of no less than £778 10s. 5d., or rather more than half the total cost. No further expenditure, however, on account of Decennial Indexes will be required for several years to come. As pointed out last year, the continual increase in the cost of the *Journal* and *Proceedings* is a source of much anxiety, and this year a further increase has to be recorded on both accounts, £219 12s. 7d. in the former and £35 18s. 2d. in the latter case, or a net increase over the cost of both in 1904 of £255 10s. 9d., and over that in 1903 of £578 3s. 3d. The Council therefore trust that authors of papers will do their utmost to assist the Publication Committee and the Treasurer in keeping down the cost of printing as far as possible.

The publication of the *Annual Reports on the Progress of Chemistry* has necessarily added materially to the expenditure of the Society; the cost of Volume I. having entailed an expenditure of £445 19s. 6d., of which about £70 has been recovered by their sale. Another item of some magnitude, £52 9s. 6d., is the cost of the printing and circulating of the Bye-laws, together with the changes proposed by the Council, which were submitted to an Extraordinary General Meeting on February 8th, 1905.

The administrative expenses have been carefully kept in check, and whilst everything has been maintained in a state of thorough efficiency their amount is only £827 16s. 5d., as against £893 1s. 0d. in 1904.

The following facts with regard to the cost of the Decennial Indexes may be of interest. The total cost of Volume IV., 1893-1902 (Parts I. and II.), exclusive of distribution, amounts to £1454 5s. 6d., of which the cost of printing was £762 6s. 0d. The cost of Volume III., 1883-

1892 (issued in one volume) was £1280 2s. 7d., of which the printing accounted for £585 19s. 7d. The cost of Volume IV. was relatively considerably less than that of Volume III., as the former extended to 451 sheets for the Authors' Index and 108 sheets for the Subject Index, or 1534 sheets in all, whilst Volume III. only contained 1024 sheets. Owing to the way in which the Annual Indexes are now prepared, it is hoped that when Volume V. has to be undertaken, the relative cost will be still further reduced and that the chief cost will be that due to the printing.

The Treasurer gave a statement as to the Society's income and expenditure during the year 1905, and proposed a vote of thanks to the Auditors, which was seconded by Dr. BERNARD DYER and carried unanimously, acknowledgment being made by Dr. H. FORSTER MORLEY.

A vote of thanks to the Treasurer, Secretaries, Foreign Secretary, and Council for their services during the past year was proposed by Dr. T. E. THORPE, seconded by Sir THOMAS STEVENSON, and unanimously adopted. Sir WILLIAM RAMSAY responded.

The PRESIDENT then delivered his Address, entitled "The Living Organism as a Chemical Agency: A Review of some of the Problems of Photosynthesis by Growing Plants."

Sir HENRY E. ROSCOE proposed a vote of thanks to the President, coupled with the request that he would allow his Address to be printed in the Society's *Transactions*. Dr. HORACE BROWN seconded the motion, which was carried by acclamation, and acknowledged by the President. (The Address will appear in the April number of the *Journal*).

The Scrutators then presented their Report to the President, who declared the following to have been duly elected as Officers and Council for the ensuing year:—

President—Raphael Meldola, F.R.S.

Vice-Presidents who have filled the office of President—

H. E. Armstrong, Ph.D., LL.D., F.R.S.; A. Crum Brown, D.Sc., LL.D., F.R.S.; Sir William Crookes, D.Sc., F.R.S.; Sir James Dewar, M.A., LL.D., F.R.S.; A. Vernon Harcourt, M.A., D.C.L., F.R.S.; H. Müller, Ph.D., LL.D., F.R.S.; W. Odling, M.A., M.B., F.R.S.; W. H. Perkin, Ph.D., LL.D., F.R.S.; J. Emerson Reynolds, Sc.D., M.D., F.R.S.; Sir Henry E. Roscoe, LL.D., F.R.S.; W. J. Russell, Ph.D., F.R.S.; T. E. Thorpe, C.B., LL.D., F.R.S.; W. A. Tilden, D.Sc., F.R.S.

Vice-Presidents—Horace T. Brown, LL.D., F.R.S.; Harold B. Dixon, M.A., F.R.S.; Rudolph Messel, Ph.D.; W. H. Perkin, jun., Ph.D., F.R.S.; A. Smithells, B.Sc., F.R.S.; W. P. Wynne, D.Sc., F.R.S.

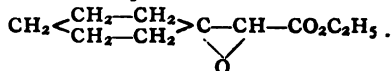
Treasurer—Alexander Scott, M.A., D.Sc., F.R.S.

Secretaries—M. O. Forster, D.Sc., Ph.D., F.R.S.; A. W. Crossley, D.Sc., Ph.D.

Foreign Secretary—Sir William Ramsay, K.C.B., LL.D., F.R.S.

Ordinary Members of Council—Edward C. C. Baly; Bernard Dyer, D.Sc.; William Gowland; Alfred D. Hall, M.A.; H. A. D. Jowett, D.Sc.; A. Lapworth, D.Sc.; J. E. Marsh, M.A.; F. E. Matthews, Ph.D.; G. T. Moody, D.Sc.; A. G. Perkin, F.R.S.; W. J. Sell, M.A., F.R.S.; John Wade, D.Sc.

Preparation of Glycidic Ethers and Aldehydes of the Hexahydroaromatic Series.—Georges Darzens and P. Lefebure.—The authors prepare from cyclohexanone and monochloroacetic ether mixed with sodium ethylate, with a yield of 65 per cent, the ether of cyclohexylacetic oxide, which has composition—



To prepare the hexahydrobenzoic aldehyde, the free acid must be slowly distilled in a vacuum, when decomposition takes place, the aldehyde and CO₂ being formed.—*Comptes Rendus*, cxlii., No. 12.

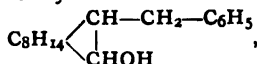
CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances de l'Académie des Sciences. Vol. cxlii., No. 12, March 19, 1906.

Distillation of Titanium and the Temperature of the Sun.—Henri Moissan.—The author finds that though the boiling-point of titanium is very high this metal can be distilled regularly. The series of experiments on the distillation of metals and non-metals lead to very interesting conclusions regarding the temperature of the surface of the sun. It is found that the maximum temperature of the electric arc is about 3500°. At this temperature all known bodies are gaseous, which result seems to point to the fact that the temperature of the sun is not above 3500°. It must be remembered, however, that these experiments are made under atmospheric pressure, and the pressure is probably very different on the solar surface.

Benzyl and Phenylborneols and their Products of Dehydration, the Benzyl and Phenylcamphenes.—A. Haller and E. Bauer.—It is known that the borneols and iso-borneols give, on dehydration, camphenes which, when treated with formic or acetic acid or in presence of sulphuric acid, give rise to the ether salts of the iso-borneols. The authors now prepare the secondary benzylborneols and the tertiary benzyl and phenylborneols, as well as their products of dehydration, the benzyl and phenylcamphenes. The secondary benzylborneols are the reduction products of benzylcamphor, and are represented by the formula—



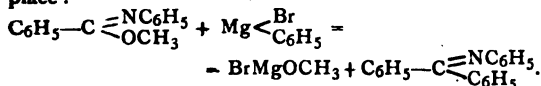
Action of Imino-ethers and Imino-chlorides on the Organo-magnesium Derivatives.—R. Marquis.—The synthesis of ketones by means of organo-magnesium derivatives has already been effected in different ways. The author solves the same problem by another method which is based on the following considerations:—The action of the ether salts on organo-magnesium derivatives gives tertiary alcohols. This reaction takes place in two phases. (a) Fixation of a molecule, $\text{R}-\text{Mg}-\text{X}$, on to the CO of the $-\text{CO}.\text{OR}'$ group. (b) A double exchange

between R and OR', resulting in the group $\begin{matrix} \text{OMgX} \\ | \\ \text{C} \\ | \\ \text{R} \end{matrix}$.

If the reaction is limited to the latter phase the group CO is blocked, and a ketone derivative results. The author therefore substitutes the imino-ethers $\text{R}-\text{C} \equiv \text{NR}'$. He first uses methyl phenylimino-benzoate,—



with magnesium phenylbromide, when under the conditions described by the author the following reaction takes place:—



Berichte der Deutschen Chemischen Gesellschaft.
Vol. xxxix., No. 4, 1906.

Crystalline Liquid Substances.—D. Vorländer.—*p*-Azoxy-benzoic acid ethyl ester is capable of existing in an anisotropic liquid phase. It has two sharp melting-points; the first, at which the solid crystalline form passes into the turbid anisotropic liquid form, occurs at 114°, and the second, at which the turbid liquid passes to the clear isotropic liquid, at 121°. Both transformations are sharp, and

occur quickly. The author has now discovered certain parallels between the azo- and azoxy-anisols, and the azo- and azoxy-benzoic acid esters. In both cases it is the azoxy-group which causes the crystalline liquid phase, for the phenomenon does not occur when they are reduced to the azo bodies. Moreover, the para-position of the substituent has an influence on the phenomenon, for *o*-azoxy-anisols forms no crystalline liquid, while the para-anisols does. The formation of the crystalline liquid is produced or favoured by the presence of the same atomic groups as those which influence other physical properties, the refraction of light, colour, rotatory power, &c. Thus all aliphatic esters of *p*-azoxy-cinnamic acid, from the methyl to the cetyl ester, have this property. Unlike the aliphatic esters, the benzyl ester of *p*-azoxy-cinnamic acid does not exhibit the phenomenon, but melts in the usual way, while the acetic acid residue and the acetophenone residue both give the anisotropic liquid and the two melting-points.

Nature of the so-called Metallic Ammonium Compounds.—Otto Ruff and Emil Geisel.—Lithium (+70°), sodium (−20°), potassium (a little above 0°), rubidium (−3°), caesium (+40°), calcium (+20°), strontium (+40°), and barium (temperature not known) at atmospheric pressure react with ammonia at the temperatures given in brackets. In the case of the alkali metals and strontium, the metal first becomes indigo-blue on its surface; the colour then rapidly changes to a beautiful metallic red (more yellowish with lithium), and then yields a solution which is an intense dark blue when very dilute. If the pressure of the ammonia above the solution is diminished, or the temperature raised, the solution loses ammonia, and coppered crystalline masses separate out. When all the liquid solution has disappeared, these masses lose ammonia, and the metal is left behind in the crystalline form. The phenomena of the crystallisation of these solutions resemble those of many saturated solutions. Calcium behaves slightly differently because it is only very slightly soluble in liquid ammonia. The authors have shown that the red masses, although their composition is represented by rMe to rNH_3 , are not chemical compounds, but that they consist of metal and liquid saturated solution. To separate solid and liquid a centrifuge was used, which, however, only partially overcame the adhesion of the solution to the solid residue. However, by packing some of the metallic ammonium in a linen cloth at a low temperature and applying pressure, the solution could be forced through the cloth, and the metal left behind. This process was not successful in the case of the caesium-ammonium substance, because it contains only 0.17 grm. of ammonia to 1.33 grm. of caesium, and it is naturally difficult to squeeze out such a small amount of liquid. The residue on the cloth, in the case of sodium and potassium ammonium, consisted of a compact piece of metal, while lithium appeared finely powdered. Rubidium and caesium caught fire on the cloth. The saturated solutions contain at −80 rNa to 5.2 NH₃, 1 K to 4.81 NH₃, and 1 Li to 3.93 NH₃. In the cases of potassium, sodium, and lithium, the solubilities were determined within as wide a temperature interval as possible, and it was proved that besides metal and saturated solution no other solid or liquid-phase could ever be detected. The solution of the alkali metals in liquid ammonia, which proceeds with exceedingly slight development of heat, is not due to transpositions such as those which occur between sulphur and liquid ammonia, but the liquids obtained are undoubtedly true solutions. The solutions of metals in non-metallic solvents seem to constitute a special type, and to possess properties marking them out from ordinary solutions. Among these characteristic properties may be mentioned their colour and their behaviour towards the electric current.

Conversion of Oxygen into Ozone at a High Temperature, and the Oxidation of Nitrogen.—Franz Fischer and Fritz Braehmer.—The authors have come to the conclusion that in order to obtain ozone by heating oxygen, the heated gases must be cooled before the ozone

has time to decompose. To do this they tried the effect of heating by various means oxygen, also as liquid air and liquid oxygen. They first investigated the phenomenon of combustion in liquid air, burning hydrogen, carbon monoxide, acetylene, &c., thus employing a high temperature caused by heat of reaction; the result of these experiments was that in the filtrate from the liquid air ozone was always found, and nitrogen oxides upon the filter except when sulphuretted hydrogen is burnt. When hydrogen is burnt in pure liquid oxygen ozone is formed, but not hydrogen peroxide. When glowing wires and Nernst pencils were used, only ozone was detected, and in the arc, and with combustion processes in liquid air nitrous anhydride was also formed. The formation of ozone is not connected with the intermediate formation of higher oxides of nitrogen, and it is undoubtedly of a thermic nature when the Nernst pencil is used, and not a photo-chemical. Possibly in the arc light some of the ozone may owe its existence to ultra-violet radiation, while in the combustion of hydrogen it is a purely thermic action. The formation in the spark is undoubtedly affected by photo-chemical action. In liquid oxygen with the Nernst pencil a solution containing nearly 1 per cent of ozone can be prepared. The following table shows the results of the experiments:—

Source of heat.	In liquid oxygen.	In liquid air.	In atmos. air.
Glowing point	Ozone	Ozone	Nitric oxide
Nernst pencil	"	"	"
Arc	"	Ozone + nitric oxide	"
H-flame	"	"	"

MISCELLANEOUS.

Iron and Steel Institute.—President, Mr. Robert A. Hadfield.—The Annual Meeting of the Institute will be held at the Institution of Civil Engineers, Great George Street, Westminster, on Thursday and Friday, May 10th and 11th, 1906, commencing each day at 10.30 o'clock a.m. The Annual Dinner of the Institute is postponed until July 27th, 1906.

Programme of Proceedings.

Thursday, May 10th—

General Meeting of Members.

The Council will present their Report for the year 1905. The Hon. Treasurer will present the Statement of Accounts for 1905.

Scrutineers will be appointed for the examination of the Voting Papers.

Election of Officers and Council.

The Bessemer Gold Medal for 1906 will be presented to Mr. Floria Osmond (Paris).

The awards of the Andrew Carnegie Gold Medal and Research Scholarships for 1906 will be announced.

Friday, May 11th—

General Meeting of the Members of the Institution of Civil Engineers.

The following is a list of Papers that are expected to be submitted:—

"The Influence of Silicon, Phosphorus, Manganese, and Aluminium on Chill in Cast-iron." By E. Adamson (West Hartlepool).

"The Influence of Manganese on Iron." By Professor J. O. Arnold (Sheffield).

"The Relation between Type of Fracture and Micro-structure of Steel Test Pieces." By C. O. Bannister, Assoc.R.S.M. (London).

"Compression of Steel Ingots in the Mould." By A. J. Capron, M.Inst.Mech.E. (Sheffield).

"The Manufacture of Rolled Solid Steel Car Wheels and Tyres." By P. Eyermann (Wisconsin).

"Brittleness in Thin Steel Sheets." By E. F. Law, Assoc.R.S.M. (London).

"Chainmaking Machinery." By E. Lelong (Couillet, Belgium).

"The Use of Oxygen in removing Blast-furnace Obstructions." By C. de Schwarz (Liege).

"Volume and Temperature Changes occurring during the Cooling of Cast-iron." By Professor Thomas Turner, M.Sc., Assoc.R.S.M. (Birmingham).

"The Influence of Copper in Steel." By F. H. Wigham (Wakefield).

The following reports on work carried out during the past year by holders of Carnegie Research Scholarships will be submitted:—

"Hardness of the Constituents of Iron and Steel." By Henry C. Boynton, D.Sc. (Cambridge, U.S.A.).

"Heat Treatment of Wire." By J. Dixon Brunton (Musselburgh).

"Quaternary Steels." By L. Guillet, D.Sc. (Paris).

"Influence of Carbon on Cast-iron." By W. H. Hatfield (Sheffield).

"The Preparation of Carbon-free Ferromanganese." By E. G. Ll. Roberts, and E. A. Wraight, Assoc.R.S.M. (London).

"Deformation and Fracture in Iron and Steel." By Walter Rosenhain, B.A., B.C.E. (Birmingham).

South African Association for the Advancement of Science.—The Meeting of the Association for the year 1906 will be held at Kimberley during the week ending July 14th, 1906, under the presidency of Mr. Gardner F. Williams. Special railway facilities will be afforded from all parts of South Africa, and arrangements are in progress for the carrying out of an instructive and interesting session. Members are earnestly desired to prepare papers for reading and discussion at the coming meeting as soon as possible, on any of the subjects in the four Sections, as follows:—(A) Astronomy, Chemistry, Mathematics, Meteorology, Physics. (B) Anthropology and Ethnology, Bacteriology, Botany, Geography, Geology and Mineralogy, Zoology, (C) Agriculture, Architecture, Engineering, Forestry, Geodesy and Surveying, Sanitary Science. (D) Archæology, Education, Mental Science, Philology, Political Economy, Sociology, and Statistics. **Competitive Essay.**—The Council have accepted with many thanks an offer from Mr. E. B. Sargent to contribute the sum of £25 for the best essay on "The Best Means of Preserving the Traditions and Customs of the Various South African Native Races in a Form available for Future Scientific Research." Papers must be received by the Assistant General Secretary, Box 1183, Johannesburg, by December 1st, 1906. Each contribution must be signed with a *nom de plume*, and the *nom de plume* accompanied by the name and address of the author (who must be a Member of the Association) must be sent in a separate sealed envelope (marked "Essay" on cover) to the same address. The Papers will be adjudicated upon by an authoritative Committee whose decision will be published as soon afterwards as possible.—E. HOPE JONES (Cape Colony and Rhodesia), FRED. ROWLAND, F.C.I.S. (Transvaal, O.R.C., and Natal), Assistant General Secretaries.

MEETINGS FOR THE WEEK.

MONDAY, 23rd.—Society of Arts, 8. (Cantor Lectures). "Ivory," by Alfred Mackell.

TUESDAY, 24th.—Royal Institution, 5. "Greek Classical Dress in Life and in Art," by Prof. G. Baldwin Brews, M.A.

WEDNESDAY, 25th.—Society of Arts, 8. "The Production and Collection of Picture Postcards," by Frederic T. Corkett.

THURSDAY, 26th.—Royal Institution, 5. "The Digestive Tract in Birds and Mammals," by Dr. P. Chalmers Mitchell, M.A.

Society of Arts, 4.30. "Seistan," by Col. Arthur H. McMahon.

FRIDAY, 27th.—Royal Institution, 9. "Ore Deposits and their Distribution in Depth," by Prof. John W. Gregory.

Physical, 5. "Some Simple Questions on the Images of Microscopes and Telescopes," by W. B. Croft. "A Gas Calorimeter," by C. V. Boys. "Lateral Vibration of Bars subjected to Forces in the Direction of their Axes," by J. Morrow.

SATURDAY, 28th.—Royal Institution, 3. "English Furniture in the Eighteenth Century," by Prof. Charles Waldstein, Ph.D., &c.

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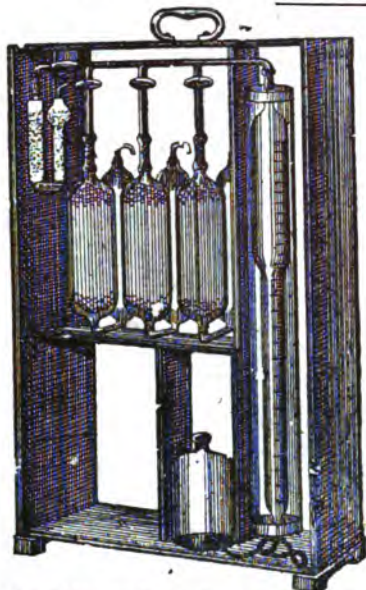
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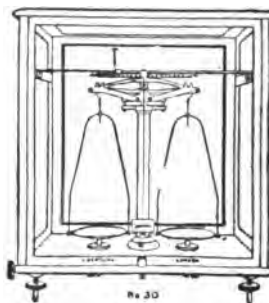
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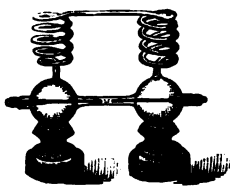
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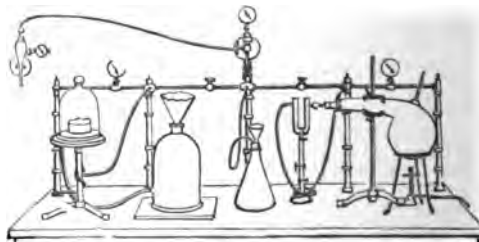
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VOL. XCIII., No. 2422.

A SYSTEM OF QUALITATIVE ANALYSIS INCLUDING NEARLY ALL THE METALLIC ELEMENTS.*

PART II.—ANALYSIS OF THE TUNGSTEN GROUP.

By ARTHUR A. NOYES.

(Continued from p. 181).

Procedure and Notes.

Procedure 31.—Dilute the HF solution (P. 6) of the residue insoluble in HNO_3 with half its volume of water, cover the dish, and pass H_2S gas into the cold solution through a platinum or lead tube. [If the solution becomes dark coloured without yielding a precipitate, add at once 1 c.c. H_2SO_4 (1·84)]. Continue to pass the gas till the solution is completely saturated. Filter through a filter supported on a platinum-ring, collecting the filtrate in a platinum dish. Heat the filtrate nearly to boiling, pass H_2S into it for five minutes, and, if a precipitate forms slowly, continue to pass the gas for at least twenty minutes, and filter again. Wash the precipitate. (Precipitate, P. 32; filtrate, P. 33).

Notes.

1. No precipitation, either cold or hot, proves the absence of antimony, arsenic, and molybdenum. An orange precipitate in the cold shows antimony; a yellow one separating from the hot solution, arsenic; a brownish black one separating from the hot solution, molybdenum.

2. From the HF solution a large part of the antimony and of the arsenic, by far the larger proportion of the molybdenum, and all of the bismuth, copper, selenium, and tellurium, are precipitated. Tin and tungsten are not precipitated even when 500 m.grms. are present, nor is a larger proportion of either of these elements carried down, even when a small quantity (2—4 m.grms.) is present together with a large quantity (300—500 m.grms.) of antimony or molybdenum. Small quantities of lead (less than 6 m.grms.) are not precipitated.

3. The HF should be cold at first, and so dilute as to contain not more than 6 per cent HF, in order to secure the separation of 1 m.grm. of antimony, which then occurs even in the presence of 500 m.grms. of tin. Though this small quantity of this element is precipitated, a large quantity does not separate completely. The H_2SO_4 is added, when the colouration shows it to be necessary, to coagulate MoS_3 , which may otherwise remain in the colloidal state, and separate upon the subsequent addition of this acid as a gelatinous mass, which is difficult to filter, and which carries down with it other elements; the addition of the acid, if not necessary for this reason, is undesirable, since it prevents the separation of a quantity of antimony less than 2 m.grms. The solution is finally heated, and again treated with H_2S in order to cause the partial separation of even 1 m.grm. of arsenic. The passing of the gas is continued so as to precipitate a large proportion of the arsenic, antimony, and molybdenum, when these are present in considerable quantity.

Procedure 32.—Heat the H_2S precipitate (P. 31) with 5—10 c.c. HNO_3 (1·20) until the sulphides are decom-

posed, adding some stronger HNO_3 (1·42) if necessary. Evaporate the solution to dryness, add HNO_3 (1·05), heat, and filter. (Filtrate, P. 51, uniting it with the main solution from P. 5; residue, reject).

Note.

1. The residue is treated with HNO_3 and the solution evaporated, so as to leave behind most of the antimony and molybdenum, the presence of which in large quantity in the main solution would somewhat increase the difficulties involved in the subsequent analysis of the H_2S precipitate. The filtrate is not likely to contain any element not also present in the main HNO_3 solution (P. 5), but it is well to unite the two solutions as a precaution.

Procedure 33.—[If it is not known from previous indications whether any element of this group is present, boil the filtrate from the H_2S precipitate (P. 31) till the H_2S is expelled, add 1 c.c. HNO_3 (1·42), evaporate nearly to dryness, pour into a platinum crucible, and evaporate to complete dryness; if there is a residue, dissolve it in a little HF].

To the filtrate from the H_2S precipitate (P. 31) [or to the HF solution of the residue after its evaporation as just described] add 2—3 c.c. H_2SO_4 (1·84), and evaporate till the acid fumes strongly. [If the liquid is dark coloured, add HNO_3 (1·42), and evaporate again]. Cool the solution, and pour it into 5 c.c. water. (Part of solution, P. 34; remainder, P. 35).

Notes.

1. A heavy yellow or greenish yellow residue proves the presence of tungsten; a blue colouration on the sides of the dish, that of molybdenum. The absence of these indications is inconclusive.

2. If SiO_2 was added to decompose fluorides (P. 3), the original residue insoluble in dilute HNO_3 (P. 5) may have consisted wholly of this substance, in which case it would be useless to continue the analysis of this group; hence the evaporation to dryness, in such a doubtful case, to see whether there is still a residue. Any SiO_2 present is volatilised as SiF_4 . HNO_3 is added to avoid volatilisation of TiF_4 , NbF_5 , and TaF_5 (P. 3, N. 4); and the solution, when nearly evaporated, is transferred to a crucible, so that a small residue may be more certainly detected. If it is doubtful whether the quantity of the residue is important; the crucible had best be weighed before re-dissolving the residue in HF, and afterwards weighed empty.

3. The evaporation with H_2SO_4 serves to expel all fluorine, which would prevent complete precipitation of the hydroxides of tantalum, niobium, and titanium at the next stage of the process, and attack the porcelain vessels used in the later operations.

4. Moderate quantities of all the elements that may be present are held in solution in the cold, diluted H_2SO_4 except tungsten. This, if present alone in quantity as large as 2—4 m.grms., separates upon dilution as a white gelatinous precipitate, or if in large amount as a heavy yellow or greenish yellow powder, even before the H_2SO_4 is diluted. Niobium and tantalum, when present in large quantity, may also precipitate, especially if the liquid is allowed to become hot.

5. If much molybdenum is present, any portions of the substance on the sides of the dish above the concentrated H_2SO_4 solution may exhibit a deep blue colour, and the diluted solution may also be deep blue, owing to partial reduction to a lower oxide. In the presence of much molybdenum, the H_2SO_4 solution may also be brown or black owing to the formation of MoS_3 after the solution was filtered; in this case it is important to add HNO_3 to re-oxidise the molybdenum, since otherwise it will not dissolve completely on the following treatment with $(\text{NH}_4)_2\text{S}$.

Procedure 34.—Filter 1 c.c. of the H_2SO_4 solution (P. 33), if not clear already, add it to 5 c.c. $(\text{NH}_4)_2\text{MoO}_4$ solution,

* From the *Technology Quarterly*, xvii., No. 3.

† When, as in this case, a variable quantity of a reagent is given, it is to be understood that the quantity used is to be proportioned within the limits indicated to the amount of the precipitate or residue.

and heat the mixture to 60° or 70° . [If a precipitate results and if H_2S produced a precipitate in the HF solution (P. 31), dilute another $\frac{1}{4}$ c.c. portion of the H_2SO_4 solution with 3 c.c. water, place it in a test-tube in a steam-bath, pass H_2S into it slowly for twenty minutes, filter, boil the filtrate till the H_2S is expelled, and add it to 5 c.c. $(\text{NH}_4)_2\text{MoO}_4$ solution]. (Precipitate and solution, reject).

Notes.

1. No precipitation proves the absence of both phosphate and arsenate. The formation of a fine yellow precipitate proves the presence of one of these radicals, and after the treatment with H_2S , it proves the presence of phosphate.

2. Arsenic acid, especially on heating to boiling, gives a yellow precipitate entirely similar to that (consisting of $(\text{NH}_4)_3\text{PO}_4 \cdot 12\text{MoO}_3$) given by phosphoric acid. If therefore arsenic can be present, as shown by the formation of a precipitate with H_2S in the HF solution, it must be first removed as described in the bracketed part of this procedure.

3. To ensure sensitiveness in this phosphate test, it is necessary that the reagent be added in large excess, so as to diminish the solubility of the precipitate, and that the solution be warmed, so as to promote the formation of the complex phosphomolybdic acid. Under these conditions 0.1 m.grm. PO_4 is detected.

Procedure 35.—To the rest of the H_2SO_4 solution (P. 33) add cautiously NH_4OH (0.90) till the solution becomes alkaline; then add cautiously 5–10 c.c. colourless $(\text{NH}_4)_2\text{S}$, and digest in a covered casserole at 50 – 60° for fifteen to thirty minutes, with frequent stirring. Filter, using suction if the precipitate is large, and wash thoroughly with hot water. [If the precipitate is still orange coloured, return it to the casserole, heat it for ten to fifteen minutes with NH_4OH (0.96) diluted with an equal volume of water, and filter again]. (Precipitate, P. 36; filtrate, P. 39).

Notes.

1. No precipitation with NH_4OH proves the absence of tin, titanium, tantalum, and niobium; none with $(\text{NH}_4)_2\text{S}$ proves the absence of the last three of these elements. An orange-red filtrate proves the presence of molybdenum; a pure yellow one, its absence. A brown filtrate indicates the presence of vanadium.

2. NH_4OH precipitates the hydrates of SnO_2 , TiO_2 , Nb_2O_5 , Ta_2O_5 , and Fe_2O_3 completely. If present alone, H_2WO_4 passes completely into solution in NH_4OH , and both it and H_2MoO_4 remain in solution upon adding $(\text{NH}_4)_2\text{S}$. The $\text{SnO}_2 \cdot \text{H}_2\text{O}$ dissolves completely upon digesting with $(\text{NH}_4)_2\text{S}$; so do any quantities of tungsten, molybdenum, or vanadium that may be present with it. When the elements that give rise to an $(\text{NH}_4)_2\text{S}$ precipitate are present, tungsten and molybdenum may be carried down with them in considerable quantity, and the latter element imparts to the precipitate an orange or red colour, which is only very slowly removed by washing, but rapidly by digestion with NH_4OH . $(\text{NH}_4)_2\text{S}$ converts FeO_3H_3 to black FeS , the presence of which often gives rise to a dark coloured precipitate. Molybdenum, even in quantity of 1 m.grm., imparts an orange-red colour to the $(\text{NH}_4)_2\text{S}$ solution; vanadium gives a yellowish brown colour.

Procedure 36.—Transfer the precipitate insoluble in NH_4OH and $(\text{NH}_4)_2\text{S}$ (P. 35), with the filter if necessary, to a casserole. Pour over it a mixture of 5–15 c.c. 3 per cent H_2O_2 and 1–3 c.c. HCl (1.12); heat till the H_2O_2 begins to decompose rapidly, stir for two to three minutes, and filter through a washed filter, using suction if the residue is large. [If the residue is large and the solution strongly coloured, dissolve the residue in HF, and treat the solution of it again by P. 33, 35, and 36, omitting only the addition of $(\text{NH}_4)_2\text{S}$]. Wash the residue. (Residue, P. 37; solution, P. 38).

Notes.

1. A yellow or orange-red colouration of the H_2O_2 solution proves the presence of titanium; a colourless solution proves its absence. A residue undissolved by H_2O_2 is not a proof of the presence of tantalum.

2. When a large quantity of titanium is present, a considerable proportion of it may remain undissolved by the H_2O_2 and HCl , and this cannot usually be dissolved by treatment with a fresh portion of the mixture. Since the presence of much titanium interferes with the subsequent tantalum test, it must be removed by solution in HF, evaporation with H_2SO_4 , precipitation with NH_4OH , and re-treatment with H_2O_2 , as directed. Moderate quantities of niobium dissolve completely, or almost completely, in the mixture of H_2O_2 and HCl . Tantalum, whether present in small or large quantity, divides itself between the residue and solution, so that each contains, roughly, one-half of it. When tin and titanium are both present in the substance in considerable quantity, the former element is retained in large quantity in the $(\text{NH}_4)_2\text{S}$ precipitate, and a residue insoluble not only in H_2O_2 , but also in strong HF, is obtained. It may be brought into solution, if desired, by fusion with Na_2CO_3 and S.

3. The HCl is added to the H_2O_2 to promote the solution of the H_2TiO_3 , and to intensify the colour produced by it.

4. If titanium is present in quantity even as small as 0.1 m.grm., the H_2O_2 solution will have a distinct yellow colour. If present in quantity as large as 100 m.grms., the solution will have a deep orange-red colour. The production of this yellow or orange-red colour constitutes the test for titanium. When it is present alone in quantity as large as 2 m.grms., and niobium is absent in considerable proportion, a confirmation is furnished later by the reddish violet colour produced when zinc is added. To determine the approximate quantity present, the colour may be compared with that given by dissolving a weighed portion of pure TiO_2 in a little HF, evaporating with 1 c.c. H_2SO_4 (1.84) to the fuming-point, adding 10 c.c. H_2O_2 , and diluting measured portions of this solution till the same colour is obtained as that of the solution to be tested.

5. Vanadium in fairly small quantity also gives an orange-yellow or orange-red colour to H_2O_2 solution; but this element rarely remains with those of the tungsten group, and the small quantity that may be present with them passes into the $(\text{NH}_4)_2\text{S}$ solution. Molybdenum gives a pure yellow colour, but it is very much fainter at the same concentration. Tungsten gives no colour.

6. The yellow or red colour of the titanium solution is probably due to the formation of a double compound $\text{TiO}_2 \cdot x\text{H}_2\text{O}_2$.

Procedure 37.—Support the filter containing the precipitate insoluble in H_2O_2 and HCl (P. 36) upon a platinum ring, and pour through it repeatedly a portion of about 2 c.c. 48 per cent HF. Add to the solution 1 c.c. HNO_3 (1.42), and evaporate it just to dryness. Dissolve the residue in 1 c.c. HF, add 0.5 grm. solid K_2CO_3 , evaporate the solution to dryness, and heat the residue until, and only until, it becomes enamel white. Dissolve it in 2–3 c.c. of water, boil the solution down to about 0.5 c.c., add 0.5 c.c. water, and allow the solution to stand for ten minutes. If a coarsely crystalline substance separates, warm the liquid slightly, add water little by little until it is dissolved, and allow the solution again to stand for ten minutes. [If a coarsely crystalline substance separates in such large quantity that more than 2 c.c. water are required for its solution, add 1 c.c. H_2SO_4 (1.84), evaporate the mixture to the fuming-point, add it to 5 c.c. water, and treat the solution by P. 35, 36, and 37]. (Precipitate and solution, reject).

Notes.

1. A heavy white or greyish translucent viscous-

seeming precipitate, but not a coarsely crystalline or light flocculent one, proves the presence of tantalum; the non-formation of such a precipitate, its absence.

2. The HF solution is evaporated before adding the K_2CO_3 , to volatilise any SiO_2 that may have been taken up from the dishes and filter-paper used, for this may cause the tantalum to remain in solution. The HNO_3 is added to prevent any loss of tantalum by volatilisation (see P. 3, N. 4).

3. When much tantalum is present, a loose, white, crystalline precipitate of K_2TaF_7 forms when the K_2CO_3 is added; for 10 c.c. of the saturated solution of this salt in water to which a very small quantity of HF is added contains only 23 m.grms. Ta. But, when it is present in small quantity, it is necessary to remove the excess of HF by evaporation and ignition, and to boil the solution of the residue as directed, in order to convert this fluoride into the far less soluble oxyfluoride ($2K_2TaF_7 \cdot Ta_2O_5$). This oxyfluoride separates as a heavy, white, translucent, viscous-seeming powder of very characteristic appearance. If the residue is too strongly heated, the tantalum separates in a lighter, more flocculent, less characteristic form.

4. K_2TiF_6 is, like K_2TaF_7 , a difficultly soluble substance, 10 c.c. of its saturated solution in water at 20° containing 25 m.grms. Ti; and this may separate at the end of the tantalum test, in case the H_2O_2 failed to dissolve out all the titanium. It is readily distinguished, however, by its appearance in the form of white, semi-transparent, granular crystals, and it is re-dissolved by gentle warming and slight dilution of the solution, which have little effect on the tantalum compound after it has once separated.

5. K_2NbOF_5 is far more soluble than the double fluorides of titanium and tantalum; thus 10 c.c. of a saturated solution of K_2NbOF_5 at 20° contains 250 m.grms. Nb. When precipitated, it resembles K_2TiF_6 in appearance. The presence of a moderate quantity of niobium does not therefore, by its separation, interfere with the tantalum test; for even if a precipitate is produced in the very concentrated solution, it dissolves on the addition of a little water. Moreover, the presence of niobium does not prevent the separation of the $2K_2TaF_7 \cdot Ta_2O_5$.

Procedure 38.—Add 1—2 c.c. H_2SO_4 (1·84) to the H_2O_2 solution (P. 36) of the $(NH_4)_2S$ precipitate, and evaporate until the H_2SO_4 begins to fume strongly. Cool completely, pour the solution into 1 c.c. HCl (1·12), add a little granulated Zn, and let the action continue for two or three minutes, noting any colour effect. Dilute the solution with 4 c.c. water, and divide it into two parts. Pour one-third of it through a column of finely granulated Zn 5 c.m. in height, contained in a small straight drying tube (1·5 c.m. in diameter) whose bulb is stuffed tightly with glass-wool, and through which previously 5—10 c.c. HCl (1·02) have been poured, to make sure that the runnings are perfectly clear. Wash out the tube with 3—5 c.c. HCl (1·02). Allow the solution and washings to run directly into 5 c.c. of nearly saturated $HgCl_2$ solution to which 0·5 c.c. HCl (1·12) has been added. Note whether a precipitate forms immediately.

If no precipitate results, pour also the remaining two-thirds of the H_2SO_4 solution through the zinc column to confirm this fact. (Solution, reject).

If a precipitate results, proceed with the remaining two-thirds of the H_2SO_4 solution as follows:—Make alkaline with NH_4OH , add a sufficient excess to dissolve the precipitated $ZnO_2 \cdot H_2O$, and filter. (Filtrate, reject). Transfer the precipitate, with the filter if necessary, to a platinum crucible, heat until the mass becomes dry and the filter is incinerated, mix the residue in a mortar with six times its volume of a mixture of equal weights of Na_2CO_3 and S, and heat the mixture in a covered porcelain crucible to a dull red heat for fifteen minutes. Cool, and treat the fused mass in the crucible with successive portions of hot water till it is disintegrated. Filter through a washed

filter and wash the residue. (Filtrate, P. 39, uniting it with the filtrate of P. 35). Transfer the precipitate, with the filter if necessary, to a platinum dish, and treat it with 5 c.c. 48 per cent HF till it is dissolved, continuing the treatment for some hours if necessary. Add 1—2 c.c. H_2SO_4 (1·84), and evaporate until the H_2SO_4 begins to fume strongly. [If the liquid is dark coloured, add HNO_3 (1·42), and evaporate again]. Cool, dilute with 5 c.c. HCl (1·02), and pour the mixture through a zinc column prepared as before, collecting the effluent in $HgCl_2$ solution. (Precipitate and solution, reject).

Notes.

1. A reddish violet colour appearing at once on the addition of Zn confirms the presence of titanium; a blue colour indicates that of niobium or tungsten. If there is no immediate precipitate in the $HgCl_2$ solution, this proves the absence of niobium; an immediate precipitate obtained in the first test shows the presence of niobium, tungsten, or molybdenum; if again obtained after the fusion with Na_2CO_3 and S, and if more than a mere turbidity, it proves the presence of niobium.

2. The solution is evaporated with H_2SO_4 to remove the H_2O_2 . It is then diluted with only a little HCl , and treated with a little Zn, so as to make the colour indications for titanium and niobium as delicate as possible.

3. If titanium is present alone in quantity as large as 2 m.grms., a distinct reddish violet colour is produced; if in quantity as large as 20 m.grms., a dark reddish violet colour results. If, however, a titanium solution containing HF—even the small quantity of it that remains after the evaporation of such a solution to slight fuming with H_2SO_4 —is treated with Zn, a green colouration is produced. If niobium is present alone in quantity as large as 0·5 m.grm., a pale blue colour results; if in quantity as large as 20 m.grms., a very dark blue colour is produced. When the reduction becomes complete, as is the case after the solution has been poured through the zinc column, a dark brown colour is obtained, even with a fairly small quantity of niobium. If titanium is present in predominating quantity, the niobium indication is obscured. Neither tantalum nor zirconium produces any colouration.

4. The salts both of titanium dioxide and niobium pentoxide are reduced to a lower state of oxidation (Ti_2O_3 and Nb_2O_3 , respectively) by the nascent hydrogen; but the lower oxide of niobium alone has sufficient reducing power to convert $HgCl_2$ into $HgCl$. The test is delicate, but not very characteristic. Neither tantalum, zirconium, titanium, iron, nor tin produces the same result under the conditions of the experiment; but titanium in large quantity may do so if the $HgCl_2$ solution is allowed to stand, even for fifteen minutes. Tantalum or zirconium is not reduced at all by Zn, and tin is reduced to the metallic state. Iron is converted into a ferrous salt, but this does not reduce $HgCl_2$. The lower oxides of tungsten, molybdenum, and vanadium (WO_2 , Mo_2O_3 , and V_2O_2) produced by the action of Zn do, however, cause reduction of $HgCl_2$; moreover, these elements are retained in considerable quantity in the $(NH_4)_2S$ precipitate, when much titanium is present. If a precipitate forms in the $HgCl_2$ solution, it may therefore arise from one of these elements and not from niobium; and it is then necessary to remove them from the remaining two-thirds of the H_2SO_4 solution by fusion with Na_2CO_3 and S, and again apply the reduction test. Repeated digestion of the $(NH_4)_2S$ precipitate with NH_4OH and $(NH_4)_2S$ is much less effective. Even after the fusion with Na_2CO_3 and S, traces of tungsten may remain in the titanium residue if much tungsten was present in the solution treated with $(NH_4)_2S$; and a slight turbidity (not more than that given by 1 m.grm. Nb) may result therefrom in the second test with Zn and $HgCl_2$. In such a case, a very slight turbidity must not therefore be regarded as proof of the presence of niobium, but a considerable one is conclusive evidence of it. A

quantity of niobium as small as 2 m.grms. may be lost in the processes connected with the fusion, but not a larger quantity.

5. The fusion with Na_2CO_3 and S is also necessary, in order to make possible the detection of small quantities of tungsten, which are carried down almost completely with the titanium hydroxide when much of this is present. It is on this account also that the aqueous solution of the fused mass is united with the original $(\text{NH}_4)_2\text{S}$ filtrate.

(To be continued).

A REVISION OF THE ATOMIC WEIGHTS OF SODIUM AND CHLORINE.*

By THEODORE WILLIAM RICHARDS
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(Continued from p. 183).

PREPARATION OF PURE MATERIALS (continued).

Silver,

RE-CRYSTALLISED silver nitrate served as the starting point for the first specimens, and the already purified residues from the early experiments were again converted into pure silver for later use. Many samples were prepared from different sources.

The silver used in all the experiments on sodic chloride was at first purified by precipitation as chloride and then recovered by reduction of the chloride with invert sugar and sodic hydroxide. While it is true that this process is not the best for the final treatment of the silver, the precipitation as chloride is an excellent step in the preliminary purification. The invert sugar was made from a filtered solution of "rock sugar." No metals were found in it by electrolytic treatment. The sodic hydroxide used, after settling and decantation, had been electrolysed until iron was completely removed. This slow but effective way of removing foreign metals was hastened by using a rotating cathode, while a fixed anode stirred the solution. The argentic chloride was precipitated from a somewhat dilute solution, to avoid the occlusion of impurities, and must be thoroughly washed. It is best to wash at first with cold water, in order to avoid the contraction of the precipitate. The reduction was carried out in a silver dish to avoid the introduction of silicates. This precaution is not needed when electrolytic purification is to follow. In some cases, where the original material was not wholly free from suspicion, the precipitation as chloride and reduction were repeated. After reduction, the washed silver was fused upon either sugar charcoal or pure lime, before the blast-lamp, whose nozzle was scrupulously cleaned. If the large buttons of metal thus formed are not kept hot too long and are cooled in a reducing flame, they are already extremely pure. (Richards, *Proc. Am. Acad.*, 1893, xxix., 65; *Zeit. Anorg. Chem.*, 1893, vi., 98; see also *Proc. Am. Acad.*, 1903, xxxviii., 450. This work was confirmed in the latter part of the present investigation.) For ordinary work, or even for work on atomic weights of usual accuracy, its purity suffices. Nevertheless, it cannot be considered as perfectly pure, for it must contain, perhaps, 0.001 per cent of sulphur and variable traces of carbon from the illuminating gas (or else oxygen from the air), as well as an occasional trace of argentic chloride, arising from incomplete reduction. When thus cooled it is likely to contain numerous minute cells inclosing gas.

As one stage in the purification the electrolytic method used by J. L. Hoskyns Abrahall has many advantages (*Journ. Chem. Soc. Trans.*, 1892, lxi., 660). The almost pure material just described is made the anode of a cell

containing a concentrated solution of argentic nitrate prepared from the same silver. The cathode is a pure silver wire, upon which is deposited by a properly regulated current beautiful crystals of electrolytic silver. The finely crystalline powder which falls from the anode (Richards and Heimrod, *Proc. Am. Acad.*, 1902, xxxvii., 415) may be easily collected separately by placing the anode button in a watch-glass or low dish, wholly submerged in the electrolyte. In order to exclude every chance of contamination, no metal but silver may be allowed to come in contact with the electrolyte, although the danger from an immersed platinum wire, either at cathode or anode, is obviously slight. Three samples of silver were thus prepared, called respectively M, N, and P. This silver must have been free from every conceivable impurity, except about 0.02 to 0.05 per cent of the mother-liquor from which it was deposited (Richards and Heimrod, *Proc. Am. Acad.*, 1902, xxxvii., 415; Richards, *Proc. Am. Philos. Soc.*, 1903, xlii., 28; *Zeitsch. Phys. Chem.*, 1903, xli., 189). This important impurity, consisting wholly of water and argentic nitrate, can be eliminated only by fusing the silver, a matter which will soon be discussed.

Besides this very pure material used in the work on sodium, several new samples of at least equal purity were made for the even more critical work on chlorine. Three of these new preparations were made from thrice crystallised pure argentic nitrate. A portion of this nitrate was converted into argentic chloride and reduced by pure invert sugar and purified sodic hydroxide. A second portion was reduced by the invert sugar and sodic hydroxide. A third portion was reduced by ammoniac formate and ammoniac acetate as recommended by Stas ("Oeuvres," iii., 40). Some of each of these lots was deposited by electrolysis in a concentrated silver nitrate bath in the usual way (S, T, U, respectively).

Not content with these preparations—although all appeared to be equally pure—and anxious to leave no stone unturned whose lifting might disclose some new source of error, we made yet two more preparations with new precautions. For one of these, through the great kindness of Mr. Richard Pearce, of the Boston and Colorado Smelting Works at Argo, Colorado, a specimen of metal was secured whose source and history were known as far back as possible. Below is given Mr. Pearce's statement concerning it:—

"This specimen is the product of the treatment of ores mainly from Colorado, in which it occurs associated with quartz, barite, calcite, pyrite, sphalerite, with more or less copper in the form of chalcopryite, together with small quantities of arsenic, antimony, lead, bismuth, and tellurium. The silver minerals are chiefly argentite, polybasite, pyrargyrite, proustite, tetrahedrite, stephanite, and occasionally cerargyrite. A small percentage of the silver comes in copper mattes purchased in other districts, more particularly Montana, where the silver ores are much the same in character, and are smelted with copper ores, forming a matte containing about 200 ounces of silver per ton.

"The roasted ore is mixed with others, principally siliceous, and the mixture is so arranged that, when smelted, it shall yield a slag containing as near as possible 40 per cent of silica and a first matte or fusible sulphide which assays 40 per cent of copper, 400 ounces of silver, and 6 ounces of gold per ton.

"The first matte always contains a certain amount of lead, but the quantity rarely exceeds 10 per cent. The next stage in the process includes the roasting and concentration of the ore-metal, or first matte, to "white metal" containing 60 per cent of copper.

"This white metal is now ready for the extraction of the silver, which comprises the following operations:—Rough roasting; fine grinding; fine roasting for sulphate of silver by Ziervogel's process; leaching, and the precipitation of the silver on plates of copper.

"In the precipitation of the silver a certain amount of copper is found mixed with the silver in the form of

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† According to Stas, saccharose at 100° would have answered as well as invert sugar at 60°. The saccharose is, of course, inverted by the strong hot alkali ("Oeuvres," iii., 13).

cuprous oxide and of small scales and scraps of metallic copper, and a process of refining is necessary previous to melting. This copper is removed by prolonged boiling with water containing a small quantity of sulphuric acid, into which air is injected by means of a small jet of steam. Sulphate of copper is formed, which is carefully washed out of the silver. The silver is then dried and melted into bars of an average fineness of 99.9 per cent."

The silver came to us in the granulated form obtained by dropping into water, and having been prepared with unusual care, was found to be even purer than Mr. Pearce claimed. After solution in nitric acid, it was crystallised fifteen times from acid solutions with centrifugal draining. The solubility in cold nitric acid is so slight that this train of purification could be effected without serious loss. Even after the second crystallisation the salt was free from any observable trace of copper, and all other metallic impurities must have been eliminated before the last crystals were obtained. The pure argentic nitrate was dissolved in pure water and precipitated by hot ammoniac formate, prepared from pure freshly distilled formic acid and ammonia which had been collected in platinum. It is better to use pure ammoniac formate instead of a mixture of ammoniac formate and acetate as Stas did, because in the former case less carbon is included in the cells of the crystals, and moreover, that which it included occurs in a less dangerous form. Since the reaction takes place according to the equation $2\text{AgNO}_3 + 2\text{HCOONH}_4 = \text{Ag}_2 + 2\text{NH}_4\text{NO}_3 + \text{CO}_2 + \text{HCOOH}$, every 170 grms. of argentic nitrate need 46 grms. of pure formic acid (or about 42 c.c. of 90 per cent acid, with specific gravity 1.2) converted into ammoniac formate. It is necessary to take this double amount in order that the ionised hydrogen (nitric acid) may be removed by the salt of the weaker acid. The precipitation was conducted in good glass. The beautiful white precipitate was washed with the purest water, until the washings gave no Nessler test. This silver, V, although it had not been electrolysed, was as pure as any which we made, judging from the analysis. (The preparation work and analyses of this silver were conducted wholly by T. W. Richards). The method of purification just described is the most convenient and safest of all. The only improvement which might be suggested is the use of a silver dish for conducting the final precipitation. This improvement was tried in the next preparation.

Part of the silver, not electrolysed, proceeding from the earlier reduction with ammoniac formate and ammoniac acetate (see above) was dissolved in re-distilled nitric acid. The silver nitrate, once more crystallised, was dissolved in pure water in a large silver dish. Ammoniac formate was freshly prepared by passing pure ammonia gas into re-distilled formic acid. With it the silver nitrate was reduced in a silver dish, but otherwise, as described above, washed free from ammonia and dried in the same silver dish. This sample was called W.

There is no reason to believe that any of these samples of silver contained any appreciable impurity, except the traces of included mother-liquor already alluded to. It is probable that Stas's silver also was equally pure at this stage in the proceedings. The subsequent treatment of the silver, in the effort to eliminate the mother-liquor and prepare the metal for weighing, seemed to be in our experiments the only cause of difference in the quantitative behaviour of our samples, and there is every reason to believe that Stas introduced during this treatment the impurities which have rendered a revision of his work imperative.

Because of this importance of the subsequent treatment, it is necessary to record a very detailed statement of the precise operations of the present research in this particular, and to point out the differences between these operations and the methods of Stas.

Everyone must agree that fusion alone is the only safe method of eliminating wholly the liquid included in the cells of the crystals, whether these crystals be produced by electrolytic or chemical reduction. The difficulty is to find

an environment for the silver during its fusion from which it will not absorb or retain foreign substances.

The question presents a twofold difficulty, because both the solid support of the silver and the atmosphere which surrounds it must be considered. The former of these difficulties may be discussed first.

In what vessel may the silver be fused, in order to avoid contamination? Stas, in his posthumous work, recommends fusion in a cupel of basic calcic phosphate, in the flame of a blast-lamp, and subsequent ignition in hydrogen. This process, while possibly as good as he contends, may nevertheless be a dangerous one. Everyone knows the risks to a platinum crucible of igniting in it a phosphate with reducing material, and yet Stas took no steps to prove the absence of traces of argentic phosphide from the metal prepared in this way. His own observation, indeed, confirms this suspicion, for he states that silver thus fused in an oxidising flame became covered with a yellow crystalline film, which instantly disappeared in a reducing flame. The great instability of the oxide of silver affords reason for doubting his assumption that this film was an oxide; it seems more probable that it was a phosphate, produced by oxidation of silver phosphide, and decomposed by reduction to this compound again. Stas, relying entirely upon the superficial appearance of the silver and its flame test as a guide to its purity, would not have discovered the presence of phosphide.

The possibility of this danger is enough to incline the careful chemist to avoid the presence of phosphorus. As a matter of fact, Stas never used silver prepared in this way for his atomic weight researches (Stas, "Ouvres" Compl., iii., 75), and no one else seems to have used this procedure except A. Scott (*Journ. Chem. Soc. Trans.*, 1901, lxxix., 147), who fused 5 grms. of silver in this way for a single analysis. Of course, however, a single analysis is too slender a basis upon which to form any adequate judgment concerning its purity. Actuated by the preceding considerations, we did not take the trouble to test the method, preferring first to test others which gave more promise of satisfactory results.

Another possible material, which has been partially tested in previous work, is carbon. Sugar charcoal may be prepared in a state very free from metallic impurities, and would serve excellently if it were not that carbon is probably soluble to a slight extent in the molten metal. We gave this possibility a fair test, and found that when the silver is fused on a cupel of sugar-charcoal with a clean blast-lamp and moderately pure illuminating gas, it is, as a matter of fact, very pure. (Experiments 67, 68, and 71). Probably the oxidising flame used to melt the metal prevents the introduction of much carbon at first, and during the brief reducing period in which the metal must be cooled to eliminate an excess of oxygen there is not time to dissolve much carbon. Evidently, however, a process which depends upon the mutual elimination of opposing errors is an uncertain one, and hence not suitable for the most accurate work. That carbon is really dissolved was well shown by another experiment, in which a carbon boat in a porcelain tube filled with pure hydrogen was used to contain the fusing silver. The experiment yielded silver slightly less pure than the previous average; it must have contained 0.005 per cent of dissolved carbon. (Expt. 79). Of course, however, a single experiment has but little exact weight in such a case, except to show that the process is probably undesirable.

Returning from these unsatisfactory methods to the one used very early in his career by Stas, we tested once more the use of lime as a support for the fusing silver. This seemed to be an especially appropriate substance for the containing vessel, because it is so easily obtained pure, and because it is so hard to reduce that no metallic calcium could be dissolved by silver. But of course the silver must be saturated with dissolved lime, and it remained to be proved that a saturated solution of lime in fused silver is so dilute as to introduce only a wholly insignificant amount of impurity. This was easily shown by actual experiment.

Ten grms. of silver fused on lime were dissolved in pure dilute nitric acid in a platinum dish, and precipitated as sulphide by pure hydrogen sulphide gas, led in through a platinum tube. After filtration on a platinum funnel, the remaining liquid was evaporated to dryness in platinum. The minute trace of residue, probably due to traces of dust, on moistening with hydrochloric acid gave no trace of the calcium bands in a spectrometer which showed them plainly from a solution containing 0.01 m.grm. of lime in 1 c.c. Hence it is evident that less than this amount of lime was dissolved by the silver, *i.e.*, less than 0.0001 per cent, if any.

This experiment settled the question concerning the supporting substance, but of course the lime must be perfectly pure. If the lime is partially made from the nitrate, it must be very thoroughly ignited before it is used, otherwise the silver may dissolve some of the oxygen caused by the decomposition of the salt. This was shown by the careful testing of a quantity of silver fused on a new boat, which had not been thoroughly ignited. (Expts. 72 to 76). A boat in which pure silver has already been fused has an advantage in this respect, as well as because every accessible trace of reducible material must have been dissolved out of it. Of course the lime used in the present experiments was especially purified, as will be related.

It is true that even when carefully prepared a boat of lime is likely to be so friable as occasionally to cause a partial coating of the button of silver with the powdered lime, but the great surface tension of molten silver keeps this all on the surface, whence it is easily dissolved by means of the dilute nitric acid always used by us to clean the metal. Scott, who has objected to the use of lime, again on the basis of a single experiment, does not mention having taken this necessary precaution.

All the silver used in the three final series of experiments which follow was thus ignited in pure well-seasoned lime boats, capable of holding from 30 to 50 grms. at each fusion. The unglazed porcelain boats in which the lime was moulded were made on purpose for this object by the Royal Berlin Porcelain Manufactory.

The environing atmosphere around the silver during fusion next claims attention, since that, too, may vitiate molten metal. Obviously air is impossible. A vacuum seemed *a priori* to be the best means of preventing the access of impurity, but, to our surprise, we found that silver fused in a vacuum was sometimes less pure than that fused in the open flame of the blast-lamp. (Expts. 72 to 76). This puzzling circumstance was ascribed finally to the supersaturation of silver with oxygen, derived either from included argentic nitrate or from the calcic nitrate in the boat. (A trace of chlorine, which, however, could hardly have been present, would have the same effect). Silver precipitated by formiate ignited on a pure boat does not show this impurity, because free from oxidising substances; and a few minutes' ignition in pure hydrogen quickly removes in chemical fashion this supersaturation when it exists, thus purifying the silver. The amount of hydrogen which remains must be on the limit of the weighable; at least, we were unable to detect it quantitatively, because silver fused in hydrogen yielded exactly the same amount of argentic chloride as that containing no oxygen which had been fused in a vacuum. This is shown by the comparison of Expts. 77, 78, 80, 81, 83, and R. 2, with 84, 85, and R. 3. Hence Stas's conclusion that silver does not dissolve over 0.0004 per cent of hydrogen is supported, although indeed his method of preparing the silver was somewhat different from ours, and his method of distinguishing the hydrogen was somewhat doubtful.

The discovery of the residual trace of oxygen in the fused electrolytic metal, which had contained occluded nitrate, was not made until the work on sodium was finished, but fortunately three pieces of the silver used in that work still remained, so that it could be compared with the purest silver which we were able to make. The comparison was made by weighing the chloride obtainable from known weights of each sample, and the details will

be recorded in full in the chapter concerning the synthesis of this salt. For the present it is enough to say that the silver used in the work on sodium yielded 0.004 per cent less chloride than the purest silver, and therefore was assumed to contain that percentage of impurity. (The average of analyses, 65, 66, 70 was compared with the average of the final series. See *prox.*) A change of only 0.002 in the atomic weight of sodium was caused by this difference.

All the silver used in the final series of syntheses of silver chloride was free from dissolved oxygen, and three of the specimens must have been free from hydrogen also. The method of treating each is sufficiently specified in the table of results, and need not be further discussed here. The silver used in this part of the work was the purest we were able to prepare, and we know of no impurities which it could have contained, except in some cases the insignificant trace of hydrogen already mentioned.

The boat used for the fusion was made from a mixture of pure lime and calcic nitrate, and was contained in a porcelain tube closed with Hempel stoppers. The tube was heated to bright redness in a Fletcher furnace (*Proc. Am. Acad.*, 1895, xxx., 379). The resulting bars of silver were freed from the adhering lime by scrubbing with clean hard sand and successive treatments with dilute nitric acid. The argentic nitrate was dissolved away by ammonia, and many washings with water were followed by drying the bar in a hard glass tube at about 400° in a vacuum. (The silver used in Experiments R 1, R 2, and R 3 was wrapped in pure silver foil during this ignition to prevent contact with hot glass). Water might otherwise become sealed into the microscopic cavities, often present in fused silver, during the next operation of cutting into blocks. The cutting is best carried out upon a silver plaque by means of a clean steel cold chisel. The blocks thus cut were superficially etched once more with nitric acid, so dilute that a possible trace of iron could not assume the passive state, and the cleaning and drying operations were repeated. The pieces when preserved in a desiccator over potash are ready for weighing and analysis.

After the details of the analysis have been discussed, the relation of the purity of this silver to that of Stas will be made clear.

Lime.

The preparation of pure lime upon which to fuse the silver was a matter of great importance, because of the danger that any impurity in it might contaminate the silver. Its preparation was begun by dissolving marble in nitric acid, boiling with an excess of calcic hydroxide, filtering, neutralising, and twice re-crystallising the calcic nitrate. (Calcic nitrate should be re-crystallised below its transition temperature, 44°, a proceeding much facilitated by inoculation with the respective hydrate). Calcic carbonate was then precipitated with ammonic carbonate, repeatedly washed, and finally dissolved in re-distilled nitric acid.

The resulting nitrate, made alkaline with ammonia, was electrolysed to remove a suspected trace of iron, and filtered through a layer of calcic carbonate upon a Gooch crucible. It was found that ammonic carbonate, of variable composition, could be conveniently distilled with steam from its solution. With such a distillate, prepared wholly in platinum, calcic carbonate was again precipitated and well washed. A small portion of this was converted into nitrate.

Ignition of the calcic carbonate to oxide was carried out in porcelain, since platinum is slightly volatile at high temperatures, and might have contaminated the lime. A dry mixture of calcic oxide, with about a fifth its weight of powdered anhydrous nitrate, was packed into specially made large unglazed boats of Berlin porcelain. The moulded mixture when heated to redness sintered firmly together and made a chemically pure receptacle, large enough to contain about 50 grms. of silver at a time. In order to prevent adhesion of the boat to the glaze of the porcelain tube, the boat should not fit the tube very closely,

and it is well to cover the glaze under the boat with a layer of pure powdered lime.

Utensils.

Of course all the containing pieces of apparatus—dishes, funnels, crucibles, retorts, condensers, and so forth—were of platinum wherever silica was to be feared and platinum was not itself harmful. In this latter case, according to circumstances, either silver vessels or vessels of the best insoluble glass (Jena or "nonsol") or porcelain, or in some cases fused quartz, were used. In this way the greatest possible purity of the preparations was assured.

(To be continued).

THIRTEENTH ANNUAL REPORT OF THE COMMITTEE ON ATOMIC WEIGHTS. DETERMINATIONS PUBLISHED IN 1905.*

By F. W. CLARKE.

(Continued from p. 184).

Strontium.

In 1894 Richards published his determinations of the atomic weight of strontium, which were based upon analyses of the bromide. A little later he made another set of measurements upon the chloride, which did not accord sufficiently well with the bromide series. The discrepancy is now explained by the discovery that the then accepted value for chlorine was too low, and the actual work upon strontium chloride has proved to be satisfactory. The data now published by Richards (*Proc. Amer. Acad.*, xl., 603; *Zeit. Anorg. Chem.*, xlvii., 145) are given below; they represent vacuum weights, and refer to Ag = 107.93 and Cl = 35.473.

Weight SrCl ₂ .	Weight Ag.	Ratio, 100 Ag ₂ to SrCl ₂ .
4.2516	5.7864	73.476
2.4019	3.2688	73.480
3.5184	4.7886	73.475
3.0264	4.1189	73.476

Mean . . . 73.477

Hence Sr = 87.661 as compared with 87.663 from the bromide.

Copper.

Incidentally to his work on the atomic weight of tellurium, Gallo made a few electrolytic precipitations of copper in comparison with silver (*Gazz. Chim. Ital.*, xxxv., 261; atomic weight computed with Ag = 107.93). The corrected data are as follows:—

Weight Cu.	Weight Ag.	Atomic weight Cu.
0.21805	0.73937	63.66
0.27153	0.92062	63.65
0.19001	0.64571	63.52
0.39585	1.34578	63.50

Mean . . . 63.58

Cadmium.

Baxter and Hines, in re-determining the atomic weight of cadmium, have made use of the chloride (*Journ. Am. Chem. Soc.*, xxvii., 222). Both of the usual ratios were measured, and the corrected data, reduced to vacuum weights, are given below.

Ratio CdCl₂ : 2AgCl.

Weight CdCl ₂ .	Weight AgCl.	Atomic weight Cd.
5.53241	8.65356	112.475
7.77758	12.16166	112.471
8.87917	13.88344	112.481

Mean . . . 112.476

* From the *Journal of the American Chemical Society*, xxviii., No. 3.

Ratio CdCl ₂ : 2Ag.		Atomic weight Cd.
Weight CdCl ₂ .	Weight Ag.	
4.92861	5.80063	112.463
3.86487	4.54891	112.454
5.08551	5.98569	112.451
5.84335	6.87704	112.468
5.99952	7.06084	112.468
3.73092	4.39095	112.467

Mean . . . 112.462

The calculations were made with Ag = 107.93 and Cl = 35.473. Additional data are promised relative to cadmium bromide. The mean of the two series, as given by Baxter and Hines, is Cd = 112.469.

Aluminium.

The work of Kohn-Abrest on the atomic weight of aluminium was noticed in the report for 1904; it has since been published in detail (*Bull. Soc. Chim.*, [3], xxxiii., 121). Impure aluminium, but containing known impurities, was dissolved in hydrochloric acid, and the hydrogen evolved was burnt over copper oxide and weighed as water. The following uncorrected amounts of water were furnished by 100 parts of the metal.

98.08
98.20
97.86
98.10
98.44
98.03
97.98

The mean, corrected for the impurities in the aluminium, is 99.151. Hence Al = 27.05 when H = 1; with O = 16, Al = 27.25.

Two additional experiments were made upon the conversion of aluminium into oxide, as follows:—

0.3429 Al gave 0.6444 Al₂O₃; Al = 27.09.
0.4168 Al gave 0.7850 Al₂O₃; Al = 27.03.

The degree of concordance shown by these determinations is not satisfactory; neither were the methods by any means unimpeachable.

Silicon.

The determination of the atomic weight of silicon by W. Becker and J. Meyer (*Zeit. Anorg. Chem.*, xliii., 251) was effected through the conversion of silicon tetrachloride into the dioxide. The chloride was decomposed by ice-water under suitable precautions, which are fully described. After decomposition, the mixture was evaporated to dryness and the silica was ignited and weighed. In a supplementary investigation by Meyer (*Zeit. Anorg. Chem.*, xlvii., 45) the possible retention of chlorine by the silica was carefully examined, and that impurity was proved to be absent. The final data, referred to vacuum weights, are given below, as published in the second of the two papers. The atomic weight was computed with Cl = 35.470.

Weight SiCl ₄ .	Weight SiO ₂ .	Atomic weight Si.
4.16733	1.47597	28.259
4.69585	1.66304	28.253
4.91918	1.74204	28.248
5.37434	1.90349	28.260
5.93985	2.10364	28.254
6.73605	2.38570	28.257
7.16361	2.53606	28.220
7.82779	2.77242	28.259

Sum . . 46.82400 16.58236 Mean 28.251

The value adopted by Meyer is Si = 28.25. This is lower than the 28.4 derived from Thorpe and Young's analyses of the bromide.

Lead.

In an interesting paper upon the lead voltameter, Betts and Kern give two series of determinations of the electrochemical equivalent of lead (*Trans. Amer. Electrochemical Soc.*, vi., 67). The metal was deposited in direct electrolytic comparison with silver from an acid solution of lead fluosulfate. The weights deposited and the corresponding equivalents were as follows, the atomic weight of silver being taken as 107.93.

Weight Ag.	Weight Pb.	Equivalent Pb.
<i>First Series.</i>		
5.8958	5.6221	103.90
5.8958	5.6396	103.24
5.7863	5.5246	103.04
5.7863	5.5450	103.43
7.8408	7.5108	103.39
7.8408	7.5168	103.47
7.6253	7.3191	103.59
7.6253	7.3221	103.62
6.2287	5.9600	103.27
6.2287	5.9605	103.28
16.6804	15.9996	103.52
16.6804	16.0014	103.54
6.8652	6.5815	103.47
6.8652	6.5812	103.46
9.3253	8.9390	103.46
9.3253	8.9419	103.49
6.8566	6.5695	103.41
6.8754	6.5877	103.41
<i>Second Series.</i>		
9.0470	8.6678	103.41
9.0470	8.6663	103.39
13.4113	12.8607	103.49
13.4113	12.8558	103.46
7.2780	6.9716	103.39
7.2780	6.9755	103.44
7.2738	6.9695	103.41
7.2738	6.9698	103.42
6.5278	6.2550	103.42
6.4864	6.2168	103.44

The mean of the determinations in the first series gives an equivalent of 103.39, or Pb = 206.78; from the second series Pb = 207.86. As the object of the investigation was to ascertain the availability of the lead voltameter for exact work, and not to determine an atomic weight, these values cannot be utilised directly. They show, however, some promise, and indicate that the process used might be developed into a good method of determination. The suggestiveness of the data warrants their reproduction here.

(To be continued).

Industrial Preparation of Calcium Hydride.—Georges F. Jaubert.—Finely-divided metallic calcium, as M. Moissan has shown, when heated absorbs a molecule of hydrogen, giving a hydride corresponding to the formula CaH_2 . This hydride, under the action of water at ordinary temperatures, is decomposed in the same manner as calcium carbide, giving a brisk evolution of pure hydrogen, $\text{CaH}_2 + 2\text{H}_2\text{O} = \text{Ca}(\text{OH})_2 + 2\text{H}_2$. The author devises an industrial preparation for this new product. The metallic calcium is prepared by electrolysis of melted calcium chloride. The electric energy necessary to prepare 100 kilogrms. of metallic calcium in twenty-four hours is about 20 volts and 7500 amperes. The hydride is manufactured from this by heating the metallic calcium in spiral vessels kept at a high temperature. Round these circulates a current of gaseous hydrogen, which the calcium gradually absorbs. After some hours of heating, all the calcium is transformed into the hydride.—*Comptes Rendus*, cxlii., No. 13.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, April 5th, 1906.

Prof. R. MELDOLA, F.R.S., President, in the Chair.

THE PRESIDENT announced the donation by Mr. John Spiller of a Berzelius medal in selenium which had been in the possession of the donor during the past fifty years.

It was also stated that the Council had appointed the following Committees:—

Finance Committee—E. G. Hooper, G. T. Moody, H. T. Brown, T. E. Thorpe, W. A. Tilden, and the Officers.

House Committee—W. R. Dunstan, J. E. Reynolds, W. J. Russell, J. M. Thomson, T. E. Thorpe, W. A. Tilden, and the Officers.

Library Committee—E. C. C. Baly, B. H. Brough, F. D. Chattaway, B. Dyer, A. Harden, C. A. Keane, A. Lapworth, E. J. Mills, J. Millar Thomson (Chairman), J. A. Voelcker, J. Wade, the Editor, and the Officers.

Publication Committee—H. E. Armstrong, E. C. C. Baly, B. Dyer, E. J. Mills, W. A. Tilden, J. Wade, and the Officers.

Research Fund Committee—H. T. Brown, W. R. Dunstan, P. F. Frankland, A. D. Hall, G. Matthney, R. Messel, H. Müller, W. H. Perkin, jun., S. P. U. Pickering, W. A. Tilden, and the Officers.

Messrs. T. V. Barker, and I. B. Hobsbaum were formally admitted Fellows of the Society.

Certificates were read for the first time in favour of Messrs. Percy Corlett Austin, M.A., Queen's College, Galway; Lionel John Drinkwater, 27, Stokenchurch Street, Fulham, S.W.; Bernard Flurscheim, Ph.D., Heatherlands, Fleet, Hants; Edgar Percy Hedley, 6, Royal Terrace West, Kingstown, Co. Dublin; Arthur Edwin Hill, 236, Willesden Lane, Brondesbury, N.W.; Frederick Gowland Hopkins, M.A., M.B., D.Sc., F.R.S., Stafford House, Newnham, Cambridge; Thomas Macdonald, North Frodingham, Driffild, Yorkshire; Harold Lawson Pendlebury, 4, Wentworth Street, Bolton; Percy George Pennymore, Eskbank Iron and Steel Works, Lithgow, N.S.W.; Walter Hansen Rawles, 81, Lewisham High Road, S.E.; John Senior, Sizing Hill, Batley Carr, Dewsbury; Emanuel George Stirmer, Park House, Margery Park Road, Forest Gate, E.; Richard Lumley Treble, B.Sc., The School Lodge, Cranbrook, Kent; Robert Hutchison Turnbull, Messrs. MacAndrews and Forbes Co., Smyrna, Turkey-in-Asia; Charles Wightman, 43, Portland Place, W.

Of the following papers, those marked * were read:—

*67. "An Improved Apparatus for Measuring Magnetic Rotations and obtaining a Powerful Sodium Light." By WILLIAM HENRY PERKIN, sen.

The author dealt with the advantages and disadvantages of the ordinary electro-magnet with pierced pole-pieces, and also the long coil or helix as a means of obtaining a magnetic field suitable for the measurement of magnetic rotations, and the desirability of obtaining an apparatus which shall possess all the advantages without the disadvantages of either of these systems. This has been effected by using a short but very powerful coil and a powerful electric current. The coil is cased with steel and has a three-inch gun-metal tube through the centre, the interior of this being the position of the magnetic field. The glass measuring tubes are supported in this tube in a metal trough which can be kept at any required temperature. Reference was also made to the measurement of the plane of polarisation and the difficulties which arise from the fact that the sodium flame is not purely monochromatic. This defect was especially felt when measuring large rotations, and was successfully

overcome by the use of a direct vision prism attached to the eye-piece or placed in the telescope of the analyser; through this a distinct image of the half-shadow disc is observed; this disc is on the analyser in the apparatus used: the prismatic colours arising from the impurity of the sodium flame are seen on either side of the image. Lastly, a method of obtaining a powerful sodium light was described, which consists in employing a large Bunsen burner with a fine tube up the centre which is supplied with oxygen. This burner, placed under a platinum-boat containing sodium chloride, is heated by the ordinary flame, which keeps the sodium chloride in a state of semi-fusion, whilst the internal flame, produced by the oxygen passing up the small tube, impinges on the side, and at the same time somewhat under the boat; this causes the sodium chloride to volatilise, and at the same time the part of the flame passing up the side receives the sodium chloride vapour, and gives a very intense yellow light, which can be maintained for a long time.

DISCUSSION.

After referring to the fact that Dr. Perkin had this year been fifty years a Fellow of the Society, Prof. ARMSTRONG expressed the opinion that probably in days to come, when physicists understood sufficient chemistry to appreciate Dr. Perkin's work on magnetic rotation, it would be regarded as of greater importance even than his discovery of the coal-tar colours.

The PRESIDENT, in moving a vote of thanks to Dr. Perkin, congratulated him on having brought to its present state of perfection the beautiful method of exploring chemical constitution by an optical method which he had conceived twenty-five years ago, and which he had practically made his own. The apparatus described was unfortunately not an ordinary laboratory installation, and he was therefore afraid that Dr. Perkin was likely to bring more work upon himself, since the discoverers of new compounds would more and more have to depend on him for the determination of magnetic rotations when, as was certain to be the case, the value of the method became more widely recognised. The President reminded the meeting how largely the pages of the *Transactions* already bore testimony to the value of the method and to the courtesy of the author in placing his services at the disposal of investigators.

*68. "The Rusting of Iron." By GERALD TATTERSALL MOODY.

Contrary to the statement of Dunstan, Jowett, and Goulding (*Trans.*, 1905, lxxvii., 1548), it is found that when bright iron is left in contact with air and water, both of which have been completely freed from carbonic acid, the metal remains untarnished, whatever the duration of the exposure. No absorption of atmospheric oxygen by iron takes place in presence of water, provided that carbonic acid has been first removed from both air and water, but on admission of a small quantity of the gas, rapid oxidation of the metal and diminution in volume of the oxygen are observed.

It is found, moreover, that the composition of iron rust is not fairly represented by the formula $\text{Fe}_2\text{O}_3(\text{OH})_2$, as stated by the foregoing investigators. An examination of a number of recently formed specimens shows that a large proportion of the iron is invariably present in the ferrous state, partly as oxide and partly as carbonate.

The explanation of rusting as a process involving the production of hydrogen peroxide, as advanced by Dunstan, is directly negated by experimental evidence, which shows that atmospheric corrosion results first from the interaction of iron and carbonic acid, whereby ferrous salt is formed, and subsequently from the more or less complete oxidation of ferrous salt by oxygen.

DISCUSSION.

Prof. ARMSTRONG said that, having seen the work, he was able to testify to the extreme care and patience which Dr. Moody had devoted to the inquiry; the experiments

had been made without bias, solely with the object of ascertaining whether iron was oxidisable by water and oxygen alone. The result was beyond question. It was not creditable to chemists, however, that problems of such fundamental importance should have so long been neglected in favour of work of no particular value to anyone, and that it should still be possible to dispute on such questions. Notwithstanding all the talk about ions, we were still without any clear accepted doctrine as to the conditions which determined the attack of oxygen on metals. Personally he had never been in doubt since he had realised that all such actions were essentially electrolytic—he had therefore felt unable to believe in the possibility of the interaction of iron, oxygen, and water taking place in the absence of an electrolyte—water not being an electrolyte. He had also never been able to accept the peroxide explanation; even supposing that rusting were inhibited by all substances which decomposed the peroxide, there was no reason to suppose that the destruction of the peroxide could in any way prevent the oxidation of the metal; some other explanation must be sought for the stoppage of action.

Mr. A. R. LING recounted his experiences with an acid water, derived from the red sandstone of Somersetshire, which vigorously attacked iron, producing rust. This action was prevented by adding to the water 7 grains per gallon of lime, but an equivalent amount of sodium carbonate was without effect. Caustic alkalis in equivalent amount prevented rusting only so long as the water was kept out of contact with the atmosphere. Rusting occurred also in presence of alkali sulphites.

Dr. GOULDING pointed out that the hydrogen peroxide solutions employed in Dunstan, Jowett, and Goulding's experiments did not contain free mineral acid, as Dr. Moody had stated, but were purified in all cases. The method usually adopted was to shake the solution thoroughly with barium carbonate, and afterwards to boil it under reduced pressure in order to eliminate carbon dioxide.

Mr. W. A. DAVIS referred to the opinion expressed by Prof. Dunstan and his colleagues in their paper, that Dr. Moody had "misread" the account given by Giorgis (*Gazzetta*, 1891, xxi., 510) of the action of hydrogen peroxide on magnesium. The tenor of this paper is to show that hydrogen peroxide when freed as far as possible from carbon dioxide attacks magnesium very much less readily than ordinary hydrogen peroxide containing carbon dioxide. Dr. Moody's statement that "hydrogen peroxide when carefully freed from carbon dioxide is *practically without action* on magnesium," is but a paraphrase of Giorgis' own conclusion that pure magnesium "è ossidato in minima quantità dall'acqua ossigenata neutra." Giorgis' hydrogen peroxide was not strictly neutral, but very slightly acid (*appena acida*), a fact which perhaps accounts for there being any action at all.

Dr. H. BORNS said that Dr. Moody had certainly taken great pains to settle the problem definitively, but he should however, have prevented the possibility of access of ozone, nitrogen compounds, sulphurous acid, &c., when finally admitting the air.

Dr. MOODY, in reply, said that the method of purifying hydrogen peroxide, as detailed by Dr. Goulding, was obviously inadequate, since in removing one impurity others were introduced. He doubted if acid could be completely removed by such a process without decomposing the whole of the hydrogen peroxide in the solution. With regard to Dr. Borns' remark, the arrangements for scrubbing the air were such that all acid constituents likely to attack iron were eliminated.

*69. "The Estimation of Carbon in Soils." By ALFRED DANIEL HALL, NORMAN HARRY JOHN MILLER, and NUMA MARMU.

Wet combustion with chromic acid has been frequently used for the estimation of carbon in soils, but, as Warrington has pointed out, it always gives results too low by 10–20 per cent. This is due to the oxidation of the carbon com-

pounds not proceeding so far as carbon dioxide, and is shown by the authors to be obviated by the introduction of a short length of red-hot copper oxide between the reacting flask and the alkali used for absorbing the carbon dioxide. The authors employ a Reiset tower for absorbing the carbon dioxide, which is then estimated by double titration, as suggested by Brown and Escombe.

DISCUSSION.

Dr. VOELCKER thought the process described would be a good one, and it had the advantage that both the carbonic acid and the organic carbon could be estimated in the one portion of soil. It would be interesting to know whether the authors had made a comparative test by Pettit and Schaub's method (*Journ. Am. Chem. Soc.*, 1904, xxvi., 1640), which consisted in combustion with sodium peroxide.

70. "Electrolysis of Salts of $\beta\beta'$ -Dimethylglutaric Acid." By JAMES WALKER and JOHN KERFOOT WOOD.

Sodium ethyl $\beta\beta'$ -dimethylglutarate, when electrolysed in concentrated aqueous solution, yields as chief product the diethyl ester of $\beta\beta\beta'\beta'$ -tetramethylsuberic acid. This ester, the corresponding acid, and some of its salts are described.

Sodium $\beta\beta'$ -dimethylglutarate under like conditions yields at the anode chiefly carbon monoxide and carbon dioxide. There is formed simultaneously, however, a small quantity of a pentene, C_5H_{10} , which was proved to be unsymmetrical methylethylethylene. The formation of this compound can only be explained on the assumption of the migration of a methyl group. This affords an analogue to the production of ethyl isolauroionate on the electrolysis of sodium orthoethylcamphorate.

71. "Bromo- and Hydroxy-derivatives of $\beta\beta\beta'\beta'$ -Tetramethylsuberic Acid." By JOHN KERFOOT WOOD.

Perkin and Thorpe (*Trans.*, 1899, lxxv., 48) found that the bromo-esters of $\beta\beta$ -dimethylglutaric acid when heated with alcoholic potash, lose hydrogen bromide, and are converted into the caronic acids. The author has investigated the action of alcoholic potash on the bromo-derivatives of $\beta\beta\beta'\beta'$ -tetramethylsuberic acid, the ester of which is the direct synthetical product obtained during the electrolysis of sodium ethyl $\beta\beta$ -dimethylglutarate. The action in this case is of a different nature from that investigated by Perkin and Thorpe; no cyclic compounds were formed, the only products isolated being hydroxy-derivatives of $\beta\beta\beta'\beta'$ -tetramethylsuberic acid.

72. "Some New *o*-Xylene Derivatives." By GEORGE STALLARD.

A new bromo-*o*-xylene ($CH_3:CH_3:Br = 1:2:3$) is obtained by hydrolysis of a bromosulphonic acid obtained by Kelbe by brominating *o*-xylene-4-sulphonic acid.

On sulphonation, it gives a mixture of two acids, one of which, the major product, has its substituents in the following positions: $-CH_3:CH_3:Br:SO_3H = 1:2:3:6$, as on debromination the *o*-xylene-3-sulphonic acid is obtained from it. The substituents of the other acid are present in the positions $CH_3:CH_3:Br:SO_3H = 1:2:3:4$ or $1:2:3:5$, as the compound is identical with Kelbe's acid mentioned above, and isomeric with the acid $CH_3:CH_3:Br:SO_3H = 1:2:4:5$, obtained by Jacobsen by sulphonating 4-bromo-*o*-xylene.

73. "A New Solvent for Gold." (Preliminary Note). By JAMES MOIR.

The author finds that gold-leaf dissolves fairly readily when floated on an acid solution of ordinary thiocarbamide. The action is rapid when a suitable oxidising agent is added; for example, when a solution of thiocarbamide is acidified and treated with a little ferric chloride, potassium dichromate, or hydrogen peroxide, the mixture dissolves gold-leaf after less than a minute's shaking. The solution

is not precipitated either by ferrous sulphate or by stannous chloride (except after long standing), whence it follows that the gold is present in solution as part of a complex ion. The gold compound has been isolated in brilliant, colourless, six-sided lozenges, striated parallel to the longest axis, and is identical with a compound which the author has obtained by boiling a mixture of sodium aurichloride and thiocarbamide solutions. It is apparently different from the compound $AuCl_2 \cdot 2CH_4N_2S$, described by Emerson Reynolds and by B. Rathke as being obtained in the reaction between aurichloride and thiocarbamide, since the mean of four concordant analyses gave $Au = 45.42$, whereas $AuCl_2 \cdot 2CH_4N_2S$ requires 51.24 per cent. Both Reynolds and Rathke give the theoretical value for gold in this compound as about 51.05, owing to the use of an incorrect value for the atomic weight of gold.

74. "The Molecular Condition in Solution of Ferrons Oxalate." (A Correction). By SAMUEL EDWARD SHEPPARD and CHARLES EDWARD KENNETH MEES.

In a paper on this subject (*Trans.*, 1905, lxxxvii., 189), the authors found for the dissociation-constant $\frac{[Fe(C_2O_4)_2]}{[C_2O_4]}$

the value 0.81 at 20°. Abegg and Schäfer working at 25° find the value 0.37, a discrepancy too considerable to be due to the temperature difference. Prof. Abegg, who has very kindly brought this circumstance to the authors' notice, has also pointed out the cause of this discrepancy. This was due to an error in the authors' calculation, whereby in calculating the concentration of $Fe(C_2O_4)_2$ the permanganate titre for C_2O_4 was added. The correct calculation is as follows:—The increase in the permanganate titre being 68.4 c.c., of this two-thirds are equal to 45.7 molecules of FeC_2O_4 dissolved. These 45.7 molecules of FeC_2O_4 , taking 45.7 molecules of C_2O_4 from the 197.8 molecules of C_2O_4 originally present, form 45.7 molecules of $Fe(C_2O_4)_2$ and leave $197.8 - 45.7 = 152.1$ molecules C_2O_4 , so that $K = \frac{45.7}{152.1} = 0.30$.

From the accepted results with longer time of stirring, the re-calculated value for K is 0.39 at 20°, in good agreement with Abegg and Schäfer's value 0.37 at 25° (*Zeit. Anorg. Chem.*, 1905, xlv., 317).

75. "Acetyl and Benzoyl Derivatives of Phthalimide and Phthalamic Acid." By ARTHUR WALSH TITHERLEY and WILLIAM LONGTON HICKS.

Phthalic anhydride, like succinic anhydride (*Trans.*, 1904, lxxxv., 1679), interacts with sodium benzamide, as well as sodium acetamide, yielding the corresponding acylphthalamic acids, $CO_2H \cdot C_6H_4 \cdot CO \cdot NH \cdot COR$. The latter readily undergo internal condensation on treatment with acetyl chloride, giving the acylphthalimides, $C_6H_4 \cdot \begin{smallmatrix} CO \\ \diagup \quad \diagdown \\ N \cdot COR \end{smallmatrix}$, identical with products obtained by the acylation of phthalimide, namely, benzoylphthalimide by pyridine benzoylation and acetylphthalimide, which has already been described by Aschan (*Ber.*, 1886, xix., 1400), by heating phthalimide with acetic anhydride.

Both acylphthalimides decompose on cautiously warming with aqueous sodium carbonate, the imide ring undergoing hydrolytic rupture with formation of the corresponding acylphthalamic acid $CO_2H \cdot C_6H_4 \cdot CO \cdot NH \cdot COR$. Aschan, on the other hand, stated that acetylphthalimide was hydrolysed with alkalis, giving phthalimide and acetic acid. The authors cannot confirm this observation, but show that when hydrolysis occurs it is preceded by rupture of the cyclic imido-grouping. Similar observations have been made by one of the authors (*Trans.*, 1904, lxxxv., 1686) in the case of benzoylsuccinimide, which on hydrolysis first yields benzoylsuccinamic acid, and finally succinic and benzoic acids and ammonia.

(To be continued).

SOCIETY OF CHEMICAL INDUSTRY.
(LONDON SECTION).

Ordinary Meeting, April 2nd, 1906.

Mr. A. G. SALAMON in the Chair.

"*Ropiness in Flour and Bread: its Detection and Prevention.*" By E. J. WATKINS.

Breads most frequently attacked by this disease are such as contain bran or low-grade white flours.

In the present investigation it has been sought, by means of culture experiments and the artificial production of ropiness in sound flour, to establish the identity of an organism isolated from specimens of rosy bread and flour obtained in England.

Cultures made from this bread yielded a small motile bacillus, which, after repeated sub-culturing, was used in a series of experiments made with a known sound flour. Varying proportions of the culture were added to the water used for making dough. Such doughs when fermented showed no sign of bacterial effects, and the bread produced was of normal character when it left the oven. The bread, when kept in a moist atmosphere at temperatures of 30–35° C., became rosy in about twenty-four hours; when the temperature was kept below 18° C., the disease did not appear. Dryness of the air generally prevented ropiness, even when the temperature was high.

Acids exercise a powerful influence in preventing the growth of the bacillus; it being found, in a series of tests with varying quantities of acetic acid in the dough, that the bread did not become rosy when kept long periods under conditions suitable to the bacillus.

The cultural and microscopic characters prove the organism to be *Bacillus Mesentericus* (Flügge).

Bakers have been held responsible for the appearance of rope in their bread, but there is good reason for the view that flour is the medium conveying the bacillus or its spores.

The great heat-resisting power of the spores of this bacillus prevents the sterilisation of the bread by baking. Flour which has been found to contain the bacillus should be held over until the cold season and then worked up with addition of a small amount of acetic acid to the dough; no fear need then be entertained of the bread being attacked during storage.

"*The Röse-Hersfeld and Sulphuric Acid Methods for the Determination of the Higher Alcohols.*" (A Criticism). By V. H. VELEV, F.R.S.

The two methods generally adopted for the determination of the higher alcohols are the Röse-Hersfeld (officially recognised in this country, Germany, and Switzerland) and the sulphuric acid method adopted in France, consequently practised in this country, and officially used as a general qualitative test for the purity of all kinds of alcohol in Russia.

Since these methods give very divergent results in the hands of different analysts, the author records various experiments to determine the accuracy or otherwise of the processes, and also criticises them.

NOTICES OF BOOKS.

The Gases of the Atmosphere. By Sir WILLIAM RAMSAY, K.C.B., F.R.S. Third Edition. London: Macmillan and Co., Ltd. New York: The Macmillan Company. 1905.

THIS version of the "tale of the air," written by one who has done much to unfold it, will be read with interest by the general reader who has had no special scientific training, and it will arouse enthusiasm to read here of the early experimenters, their successes and failures, and their blind guesses after truth, all of which Sir William Ramsay

describes graphically, giving interesting extracts from the works of the early chemists who occupied themselves with the study of the air. Then the author passes to the discovery of argon and the investigation of its properties, followed by the other inactive gases, while to the third edition a chapter has been added on the radio-active gases (the emanations), in which a short and concise account is given of the discovery of radio-activity, its detection, and the law of decay, which is discussed exceptionally clearly. The investigation of the properties and behaviour of the emanations, as far as we now know them, brings to an end a book every page of which is full of interest for the chemist as well as the general reader.

Über die Vorgeschichte und die Anfänge der Chemie. ("The Early History and the Beginnings of Chemistry"). By Dr. FRANZ STRUNZ. Leipzig and Wien: Franz Deuticke. 1906.

THIS short introduction to the history of the chemistry of antiquity bears evidence of painstaking work on the part of its author, who has selected his material with the utmost care. Though only very short (it could easily be read through from beginning to end in an hour), it is full of interesting information. Only the most important of the developments of early chemistry are to be found in the book, and the subject is treated more especially from the point of view of the practice of the ancients chiefly as regards metallurgical operations. Thus, while the earliest methods of extracting the better known metals from their ores are fully discussed, we find no accounts of the manufacture of glass and soap. The introduction summarises such theoretical views on the composition of substances as were current among the older philosophers, and this chapter is particularly interesting and brightly written.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances de l'Académie des Sciences. Vol. cxlii., No. 13, March 26, 1906.

Isolation of Dysprosium, and its chief Atomic Characteristics.—G. Urbain.—The author isolates about 50 grms. of an earth which presents the characteristic spectrum of dysprosium. Four consecutive fractions have the same mean atomic weight, 162.49. The oxide is white, and does not change to peroxide when calcined in oxygen. The salts have a yellowish green coloration. The chief characteristics of the dysprosium compounds, such as solubility of salts, basicity of oxide, &c., place this element in the series of the rare earths between terbium and the new holmium. Outside the visible absorption spectrum, dysprosium presents a particularly sharp ultra-violet spectrum, composed of intense diffused bands. The whole spectrum contains the following lines, a neutral solution of chloride being used.

	λ.	
400	to 394	Weak; very diffused.
392	" 384.5	Very strong; diffused.
381.5	" 377	Maximum 379.5.
368.5	" 361.5	Very strong; diffused; maximum 365.
355.5	" 345.5	Extremely strong and diffused; max. 351.
340	" 336	Strong; diffused; maximum 338.
329	" 316	Extremely strong; maximum 322.5.

The Action of Xantic Leucomains on Copper.—N. Slomnesco.—Theobromine, theophiline, urea, and probably also the other xantic leucomains have the property of precipitating copper from its solution in the form of the yellow hydrate. The action is not only a

simple reduction but consists also of a tendency for the bases to combine with the copper. If copper is boiled with these bases in aqueous solution the same characteristic precipitate is produced, which has a dirty yellow colour.

A New Type of Equilibrium Reaction.—L. J. Simon.—The author makes a series of experiments on the equilibrium between pyruvic acid and urethane in the absence of all condensation agents. He also suspends a known weight of pyruvic acid in a definite volume of water. This rapidly dissolves when the temperature is raised. On cooling, the dissociation is measured. He finds:—1. That for a definite concentration the state of the solution depends on the temperature to which it has been carried. The dissociation increases with the temperature. 2. The dissociation increases with the concentration, on account of the higher temperature which must be attained to ensure solution in the same time. 3. The dissociation is total for the greatest concentrations. For weak solutions there seems to be one molecule dissolved to one dissociated.

Estimation of Cadmium.—H. Baubigny.—In a previous research the author proved that cadmium sulphide is not as easily decomposed as has hitherto been supposed. Under certain conditions, which the author describes in detail in the present paper, the filter-paper containing the sulphide precipitate can be burnt in the ordinary way.

Estimation of the Albumenoid Matter in Milk.—MM. Trillat and Saunton.—The authors describe a method by which (1) all the albumenoid matter can be separated from milk; (2) it can be shown to possess the elementary composition of casein; (3) it can be shown that there is no variation in weight.

MISCELLANEOUS.

Effects of Blows upon Chemical Elements, especially the Light Metals.—O. Ohmann.—1. *Calcium*.—Doermer (*Berichte*, 1906, xxxix., 211) has already described the phenomenon which occurs when electrolytic calcium is struck upon an anvil, but he observes that the explosibility is much smaller if no iron oxide or rust is present. The author, however, finds that sparks appear when the metal is struck between two pieces of quartz and of granite. He believes that a partial vaporisation of calcium takes place at the place where the pressure is greatest, and it combines with oxygen. This view is confirmed by the following facts:—(a) A brighter light is produced when the experiment is performed in an atmosphere of oxygen; (b) if the metal is struck with the edge of the hammer the sparks appear, but if the hammer is rounded, or the material is not very hard, only a faint light is seen; (c) if a sharp blow is struck with the whole surface of the hammer an explosion occurs; this appears to be an explosion more in a physical than in a chemical sense; (d) if heat has accumulated in consequence of many unsuccessful blows, sometimes after a comparatively gentle blow an intense light appears, and an explosion occurs. 2. *Sodium*.—A clear luminescence appears when a piece of sodium as big as a pea is struck with a hammer. 3. *Potassium*.—With potassium a vigorous action occurs at each blow; with very moderate blows violet flames strike out on all sides, and clouds of vapour with a piercing odour are formed. If it is struck more sharply, glowing balls are projected to some distance. 4. *Lithium*.—A very small piece of lithium after a few blows gives with each subsequent blow a bright flame often accompanied by an explosion. The flame is sometimes red or white and dazzling (especially when the blows are hard). Besides these flames small sparks appear, very much like the sparks obtained from iron. 5. *Aluminium*.—Only very small sparks were obtained when the powdered metal was struck, and sometimes no phenomena at all occurred. Electrolytic

barium would probably exhibit the phenomenon, but it does not appear with barium obtained from amalgam. Thallium gave no result. 6. *Phosphorus*.—When a few c.m. were struck a doughy plate was formed, from which sparks were obtained.—*Berichte*, xxxix., No. 4.

MEETINGS FOR THE WEEK.

- MONDAY, 30th.**—Society of Arts, 8. (Cantor Lectures). "Ivory in Commerce and in the Arts," by Alfred Maskell.
- TUESDAY, May 1st.**—Royal Institution, 5. "Greek Classical Dress in Life and in Art," by Prof. G. Baldwin Brown, M.A.
— Royal Institution, 5. (Annual Meeting).
— Society of Arts, 4.30. "Social Conditions in Australia," by the Hon. J. G. Jenkins.
- WEDNESDAY, 2nd.**—Society of Arts, 8. "Submarine Signalling," by J. B. Millet.
— Society of Public Analysts, 8. "Estimation of Fat in Homogenised Milk," by H. Droop Richmond. "Milk Analysis," by H. Droop Richmond and E. H. Miller. "Composition of Saffron," by A. E. Parkes. "The Polenske Method for the Detection of Coconut Oil in Butter," by S. Rideal and H. G. Harrison. "Presence and Detection of Cyanogen in Java and other Beans," by R. K. Tailock and K. T. Thomson. "Examination of Linseed, Olive, and other Oils," by R. T. Thomson and H. Dunlop.
- THURSDAY, 3rd.**—Royal Institution, 5. "The Digestive Tract in Birds and Mammals," by Dr. P. Chalmers Mitchell, M.A.
— Chemical, 8.30. "Relation between Absorption Spectra and Chemical Constitution—Part V. The Isonitroso-compounds," by E. C. C. Baly, E. G. Marsden, and A. W. Stewart. "Action of Tribromopropane on the Sodium Derivative of Ethyl Malonate" (Part II.), by W. H. Perkin, jun., and J. L. Simonsen. "Brazilin and Hamatocrylin—Part VII. Some Derivatives of Brazilin," by P. Engels and W. H. Perkin, jun. "Pipitzaholic Acid," by J. M. Sanders. "Constitution of the Hydroxides and Cyanides obtained from Acridine, Methyl-acridine, and Phenanthridine Methiodides," C. K. Tinkler. "Constitution of Ammonium Amalgam," by E. M. Rich and M. W. Travers. "Action of Light on Potassium Ferrocyanide," by G. W. A. Foote.
- FRIDAY, 4th.**—Royal Institution, 9. "The Steam Turbine on Land and at Sea," by the Hon. Charles A. Parsons, C.B., F.R.S., &c.
- SATURDAY, 5th.**—Royal Institution, 3. "English Furniture in the Eighteenth Century," by Prof. Charles Waldstein, Ph.D., &c.

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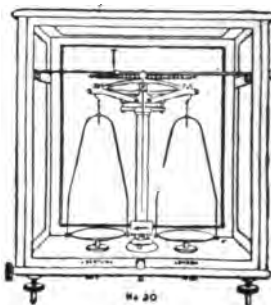
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THE CHEMICAL NEWS.

VOL. XCIII., No. 2423.

A MODIFIED EVOLUTION METHOD FOR THE DETERMINATION OF SULPHUR IN PIG-IRON.

By J. McFARLANE and A. W. GREGORY.

It is well known that only part of the sulphur in pig-iron is evolved as hydrogen sulphide by the action of hydrochloric or sulphuric acid. Pig-irons containing a considerable proportion of combined carbon (mottled and white irons, and the shots obtained by pouring the molten metal into water), when acted on by hydrochloric acid, give only a part of their sulphur as hydrogen sulphide—the proportion varying very considerably, and methods of sulphur determination based on this reaction are unreliable.

This difficulty is partially overcome by annealing the powdered sample at a bright red heat in an inert atmosphere (Walters and Miller, *Iron. and Steel Inst.*, 1902, No. 1, p. 851). If, however, the powdered pig-iron be mixed with a little cream of tartar and heated to redness in a covered crucible for some time, the whole of the sulphur may be evolved as hydrogen sulphide by the action of hydrochloric acid, and the sulphur estimated by the oxidation of this compound. The exact method of working which we have adopted is as follows:—

Five grms. of the powdered sample are taken and mixed intimately with about 0.5 gm. of cream of tartar, the mixture wrapped in filter-paper and placed in a small crucible. The crucible is covered and heated to bright redness in a muffle for a quarter of an hour, after which it is removed and allowed to cool. The resulting mass is powdered by gentle crushing in a glass mortar and transferred to the evolution flask, the delivery tube of which dips into an ammoniacal cadmium chloride solution. Boiling hydrochloric acid (2 parts acid to one part water) is then introduced into the evolution flask. When the reaction has become quiescent, the liquid in the flask is boiled to expel hydrogen sulphide from solution.

The sulphur is now in the form of cadmium sulphide. The liquid containing this in suspension is acidified with hydrochloric acid, and the hydrogen sulphide in solution immediately titrated with standard iodine solution, in the same manner as in the estimation of sulphur in steel by the cadmium chloride method.

The cadmium chloride solution is prepared by dissolving 20 grms. of cadmium chloride in a litre of water and adding a litre of strong ammonia.

For each estimation, 25 c.c. of this solution is diluted with about 300 c.c. of water and placed in a tall beaker. The iodine solution, each c.c. of which is equivalent to 0.00025 gm. of sulphur, is standardised by means of a steel of known sulphur content. As a check on our results, a series of pig-irons was taken and the sulphur in each accurately determined by the "aqua regia" method and also by the evolution process given above.

In order to ensure great accuracy in the aqua regia method, we proceeded as follows:—2½ grms. and 7½ grms. of the sample were separately dissolved in 70 c.c. of a mixture of hydrochloric and nitric acids (100 c.c. HNO₃ and 50 c.c. HCl) to which a few grms. of potassium chlorate had been added. Each was evaporated carefully to dryness and roasted, allowed to cool, taken up in 40 c.c. hydrochloric acid, evaporated to dryness again to expel nitric acid, dissolved in 50 c.c. hydrochloric acid, boiled to low bulk, diluted, and filtered.

The filtrates were diluted with 400 c.c. of distilled water, boiled, and precipitated with 50 c.c. of 20 per cent solution of barium chloride. The solutions were allowed to stand over night on a warm plate.

The barium sulphate precipitate was filtered off, washed with very dilute boiling barium chloride solution containing a little hydrochloric acid to remove the last traces of iron. Finally, the barium sulphate was washed with boiling distilled water, ignited, and weighed. The difference between the weight of barium sulphate obtained from the 7.5 grms. and the 2.5 grms. of pig-iron gave the true weight of barium sulphate derived from 5 grms.; errors due to the reagents used containing sulphur being thus eliminated. The following results were obtained by the two methods:—

	Aqua regia method. Per cent S.		Evolution method. Per cent S.	
1.	—	0.093	0.090	0.089
2.	0.090	0.090	0.091	0.094
3.	0.096	0.098	0.093	0.095
4.	0.066	0.066	0.066	0.068
5.	0.078	0.080	0.080	0.080
6.	0.060	0.060	0.060	0.061
7.	0.063	0.063	0.062	0.061
8.	—	0.076	0.078	0.078
9.	0.069	0.068	0.069	0.070
10.	—	0.082	0.080	0.080

The above results were obtained with samples broken from ordinary pigs. Equally good results were obtained with "chilled shot" samples as follows:—

	Aqua regia method. Per cent S.		Evolution method. Per cent S.	
1.	0.186	0.188	0.190	—
2.	0.197	0.204	—	—
3.	0.129	0.129	—	—
4.	0.115	0.113	—	—
5.	0.086	0.085	0.087	—
6.	0.148	0.152	—	—
7.	0.222	0.222	—	—

The following results were obtained with "chilled" samples containing a very high percentage of sulphur:—

	Aqua regia method. Per cent S.		Evolution method. Per cent S.	
1.	0.448	0.440	0.438	—
1.	0.292	0.285	0.282	—
3.	0.390	0.402	0.406	—
4.	0.476	0.458	0.464	—

The cream of tartar used should be free from sulphur (usually present as sulphates).

No elaborate absorption vessel is required, since the hydrogen sulphide is completely absorbed by allowing the evolved gases to bubble through the cadmium chloride solution contained in a beaker.

The efficiency of this apparently crude method of absorption was shown by the following experiment:—

A pig containing over 0.6 per cent of sulphur was dissolved in the evolution flask, a very rapid evolution being maintained. The evolved gases were passed through two small flasks containing the diluted ammoniacal cadmium chloride solution, first passing through No. 1 flask and then through No. 2.

There was no trace of a precipitate in No. 2 flask, and, after its contents were acidified, only 0.5 c.c. of the standard iodine solution was required to oxidise the hydrogen sulphide absorbed, equivalent to 0.00013 gm. of sulphur. The drillings or powdered pig-iron should be in a fine state of division, so that the evolution of gas may be as rapid as possible.

Technical College, Finsbury, Old Students' Association.—A Meeting of Old Students, at which Sir Owen Roberts has kindly consented to preside, will be held at the Technical College, Finsbury, on May 8th, at 8 p.m. Any old student who has not received a notice of this meeting is requested to communicate with J. W. G. Brooker, Durlstone, Brockley Park, Forest Hill, S.E.

A REVISION OF THE ATOMIC WEIGHTS OF
SODIUM AND CHLORINE.*By THEODORE WILLIAM RICHARDS
and
ROGER CLARK WELLS.

(Continued from p. 195).

THE SOLUBILITY OF ARGENTIC CHLORIDE.

MULDER pointed out in 1857 that the solubility of argentic chloride is sufficient to affect all quantitative results into which the precipitation of this substance enters ("Die Silber-Probirmethode, Leipzig, 1859). Stas overlooked this solubility in his early determinations, and hence in 1876 repeated some of them ("Oeuvres," i., 751). Other more recent experimenters have in general confirmed Stas's later conclusions about this solubility, so that it is not important here to give a lengthy statement of our further confirmatory results. (For references see Böttger, *Zeit. Phys. Chem.*, 1903, xlv., 189; Richards, *Proc. Am. Acad.* 1893, xxix., 69).

There can be no doubt that freshly precipitated argentic chloride is soluble in cold water to the extent of several m.grms. per litre, and that this solubility steadily diminishes, without a very sharp change at any time, as the precipitate remains in contact with the liquid.

A variable solubility of this nature may be due to one of two causes. Either the precipitate is capable of assuming two or more allotropic forms, of which the least stable appears at first (Ostwald, *Zeit. Phys. Chem.*, 1897, xxii., 307), or else it separates in a finely divided state, and gradually becomes aggregated into larger masses. The work of Ostwald (*Zeit. Phys. Chem.*, 1900, xxxiv., 495) and of Hulett (*Zeit. Phys. Chem.*, 1901, xxxvii., 385; 1904, xlvii., 357) has shown how greatly a difference in size of the grains may affect the solubility, especially of a salt only slightly soluble.

The latter explanation seems the more probable for several reasons. In the first place, no sharp change in the solubility has ever been detected, such as would be expected from the appearance of a new more stable solid phase. Again, the difference between the specific gravity of the flocculent precipitate and fused horn-silver is very slight, and their heats of solution in cyanide are identical, indicating little or no difference of molecular constitution between the two. (Lævinsohn determined the specific gravities; his results were verified and the thermo-chemical argument added to them by Dr. W. N. Stull and one of us, in some work not yet published). Moreover, as will be shown later, the curdy precipitate may be successfully washed in a measure unattainable with crystalline powders, showing that the innermost recesses of the solid are more than usually accessible, and therefore that the structure is very finely divided and loosely knit. The more compact, less soluble precipitate, obtained either by heating or by long standing, has largely lost this possibility of being thoroughly washed, and has become also much less soluble—facts which are quite in line with the above hypothesis. However this may be, the uncertainty of the solubility is a fact of important significance to the analyst, for it obliges him to determine the amount of solution in every experiment—the solubility can never be assumed as known.

The matter is yet further complicated by the well-known fact that the presence of excess of either ionised silver or ionised chlorine causes a depression of the solubility, in the manner demanded by the law of concentration effect (Hoitsema, *Zeit. Phys. Chem.*, 1896, xx., 272). A large excess of either precipitant reduces the solubility to so small a value as to be practically negligible in most cases (see below). This may indeed be the reason why the precipitating liquids should for more than an instant be exactly equivalent. Hence each first particle of precipitate would

quickly be surrounded by a liquid unfavourable to its union with another in compact crystalline fashion.

Mulder thought that an excess of either silver or chloride equivalent to four times the weight of the dissolved argentic chloride was enough to complete the precipitation, and Stas thought that exactly three times the equivalent amount sufficed. It is clear now that this limit is determined merely by the efficiency of the means used to detect the last faint cloud of precipitate, and then only approximately.

As has been said, the changing solubility demands that every filtrate be tested quantitatively. The methods which may be used are all open to some criticism. The determination of electrolytic conductivity is in vain when any other electrolyte is present, and its interpretation is complicated even in pure aqueous solution by possible hydrolytic complications. The determination of electromotive force depends upon the rigorous application of the law of concentration effect to electrolytes—a somewhat doubtful question. The actual evaporation of the solution and weighing of the precipitate leads to many errors when silver is in excess (Richards, *Proc. Am. Acad.*, 1893, xxix., 71), and the determination of the intensity of the opalescence upon precipitating the dissolved chloride depends greatly on the details of the production of the opalescence.

From among all these methods we chose the last, after a very careful study of their probable errors. But although fairly confident of its results, for reasons shortly to be given, we were careful in every case to allow in each experiment as little dissolved argentic chloride as possible, so as to reduce the possible error to a minimum.

The method of comparing the intensity of the cloudiness of opalescent solutions, and therefore the quantities of suspended precipitate, has already been discussed at some length in a paper on the nephelometer (Richards and Wells, *Am. Chem. Journ.*, 1904, xxxi., 235). This instrument consists of a device for the comparison of the tint or brightness of two solutions containing precipitate, viewed against a dark background. By making one of these opalescent mixtures from a known amount of material, an unknown amount may be estimated by comparison with it.

It is easy to show that with this instrument the average of many results is within 1 or 2 per cent of the truth, so that the error with 2 m.grms. of dissolved argentic chloride is probably less than 0.04 m.grm. This degree of accuracy is as great as can be obtained in the collection of a precipitate for weighing, hence the method is to be ranked among those suitable for exact work.

With the perfected nephelometer it was possible to study the progressing precipitation of very dilute argentic chloride in a way previously impossible, and several unexpected irregularities were found during its course. Since these are important in the quantitative sense, they must all be briefly discussed.

The most striking of these is the variation in the time of complete precipitation caused by the presence of electrolytes. For example, a solution of argentic chloride, on adding a slight excess of soluble argentic salt, becomes opalescent, far more quickly in the presence of much sodic nitrate or nitric acid than in the absence of such an electrolyte. At the end of five minutes the former solution appears about three times as cloudy as the latter; and several hours are needed for the two solutions to attain equality of opalescence at a maximum value, which thereafter remains constant. It appears from this fact that the precipitate probably at first forms in a colloidal state, since electrolytes are known to hasten the precipitation of colloids. This inference is confirmed by the occasional appearance of pale blue or pink tints in the incipient precipitate, which disappears as the cloudiness becomes more intense.

Oddly enough, a solution of pure argentic chloride (made by shaking the curdy precipitate with water and filtering) attains its maximum cloudiness after subsequent addition of argentic nitrate more rapidly than a solution of similar dilution made by mixing equivalent amounts of very dilute

* Published by the Carnegie Institution of Washington, April, 1905.

argentic nitrate and sodic chloride solutions. The trace of sodic nitrate left in the latter case seems to be unable to counteract some other tendency holding the precipitate in solution. At first it seemed possible that minute invisible "crystal germs" might be present in the filtered solution, which accelerated the appearance of the opalescence, but this surmise was soon overthrown experimentally. A solution made from curdy silver chloride and found nephelometrically to contain 1.28 m.grm. per litre, was diluted with an equal bulk of pure water, and its concentration after agitation for over one hour was determined by the nephelometer. The average concentration found by five trials, 0.61 m.grm. per litre, agree with that expected within a reasonable limit of error, and, moreover, the speed of precipitation was no less than before. There could hardly have been any "crystal germs" existing in the more dilute solution, hence one must assume that the undissociated part of the argentic chloride is really in a different state, when it is dissolved out of salt long formed from that in which it is when in the act of precipitation. The observation at least adds one more anomalous fact, perhaps giving a clue to some of the other anomalous facts, concerning the solubility of argentic chloride.

The application of these observations to the nephelometric analysis of a very dilute silver chloride solution is obvious. It is a difficult matter to prepare a standard which will behave in exactly the same way as the solution to be estimated. By adding appreciable quantities of nitric acid to each, it is nevertheless possible to hasten the precipitation of each so greatly as to cause them to proceed at a speed almost equal; and the maximum cloudiness thus quickly attained persists unchanged in relative intensity for hours. It seems highly probable that the result thus reached really represents the amount of dissolved argentic chloride; especially because the same result is attained after a much longer time even when no electrolyte is present.

The actual extent of the solubility of argentic chloride thus estimated varied through wide limits, as has already been stated. For the solution freshly filtered from the curdy precipitate in the usual condition at 20° the amount dissolved was ordinarily about 1.5 m.grms. per litre, a result about equal to that of Kohlrausch and Rose and others. On long standing such a solution was found to decrease in concentration, on one occasion to as little as 1.1 m.grm. per litre, although no visible precipitate was seen on the glass. The trace of material represented by this difference was probably adsorbed by the glass. This observation points out the necessity of determining the strength of an unknown solution immediately after its filtration, a precaution which was carefully heeded in the quantitative part of the work.

Although sodic nitrate and nitric acid hasten the precipitation of the curdy opaque form of argentic chloride, they augment slightly the amount which finally remains in solution. By means of the nephelometer it was found that a saturated solution of curdy argentic chloride in the presence of about tenth normal sodic nitrate and a small amount of nitric acid usually contained as much as 3 m.grms. of the slightly soluble halide per litre. A mixture of this type was present at the conclusion of each nephelometric experiment recorded in the experiment on the ratio between sodic chloride and silver, and the bearing of the actual value on the investigation will be there discussed.

The solubility of silver chloride in a liquid containing a large excess of one of its ions has been stated above to be "practically negligible in most cases." In determining the ratio between silver and silver chloride, however, it seemed advisable to determine even this solubility, which may partly represent the solubility of the undissociated silver chloride, as distinguished from that ionised, and also includes the slight correction for the imperfect retaining power of even the best filters. The fact that a single part of silver chloride may be detected in 30,000,000 parts of water by the nephelometer in the presence of an excess of

silver nitrate shows the approximate order of this solubility (Richards, *Proc. Am. Acad.*, 1893, xxix., 74).

In five experiments, in which silver chloride was precipitated by an excess of hydrochloric acid, the combined mother-liquor and wash waters remaining after filtration were evaporated to dryness. The residue was taken up with a very small quantity of ammonia, and then neutralised with nitric acid. After the addition of a slight amount of sodic chloride, the silver chloride was estimated by the nephelometer, by comparison with tubes made up with known amounts of silver in the same way.

Silver Chloride Found.

In 3 litres	0.09 m.grm. AgCl
In 3 litres	0.13 "
In 3 litres	0.05 "
In 2 litres	0.05 "
In 2 litres	0.05 "
Mean	0.03 m.grm. per litre

This value represents the solubility of argentic chloride in water containing sodic nitrate and an excess of hydrochloric acid, together with those traces of the substance which could not be collected upon a filter. It is not certain that the same value would be obtained if it were possible to determine this infinitesimal solubility in the presence of an excess of argentic nitrate; but the fact that the limiting value previously found by one of us is about of this order seems to render this probable. Hence, for the sake of completeness, the correction was applied to the results in the latter case as well as in the former, although it was so small as not even to affect the third decimal place of the atomic weight of sodium.

Further consideration of the solubility of argentic chloride will be given in other appropriate places.

(To be continued).

THIRTEENTH ANNUAL REPORT OF THE COMMITTEE ON ATOMIC WEIGHTS. DETERMINATIONS PUBLISHED IN 1905.*

By F. W. CLARKE.

(Continued from p. 196).

Tellurium.

THE electro-chemical equivalent of tellurium has been determined by Gallo, in comparison with silver taken as $Ag = 107.93$ (*Atti Accad. Lincei*, [5], xiv., 33, 104; *Gazz. Chim. Ital.*, xxxv., 245). Three series of determinations are given, with weights reduced to a vacuum. The data are as follows:—

Weight Ag.	Weight Te.	Atomic weight Te.
0.74117	0.218412	127.22
1.03801	0.304514	126.65
0.91704	0.27256	128.30
1.041101	0.307117	127.35
1.09064	0.321952	127.42
1.16302	0.34582	128.16
0.968903	0.28646	127.64
1.518712	0.44767	127.28
0.906561	0.26836	127.76
0.995511	0.29586	128.28
0.86596	0.25656	127.90
1.11282	0.328318	127.44
Mean		127.617

Although these values are widely variable, the mean approaches closely to the atomic weight as determined by other investigators.

* From the *Journal of the American Chemical Society*, xxviii., No. 3.

Gutbier's re-determination of the atomic weight of tellurium is based upon the reduction of scrupulously purified tellurium dioxide by two distinct methods (*Ann.*, cccxlii., 266). In the first series of experiments the dioxide, mixed with finely-divided silver and quartz sand, was reduced by pure hydrogen. In the second series, hydrazine was the reducing agent. The data, with vacuum weights, are as follows:—

Weight TeO ₂ .	Weight Te.	Atomic weight.
<i>Hydrogen Series.</i>		
2'99688	2'39585	127'55
1'30740	1'04527	127'60
2'04325	1'63380	127'68
2'61725	2'09249	127'59
3'61725	2'89222	127'65
Mean		127'614
<i>Hydrazine Series.</i>		
1'90601	1'52390	127'62
1'03532	0'82784	127'67
2'2200	1'77480	127'55
Mean		127'613

Gutbier thus reaches the same value for tellurium as Gallo, which may be rounded off to 127'6; this is the value adopted in the International Table.

Bismuth.

In a Doctoral Dissertation by Lothar Birckenbach, some experiments are described involving the synthesis of bismuth trioxide from the metal, and also the reduction of the oxide to metal ("Ueber das Atomgewicht des Wismuths," Inaugural Dissertation, Erlangen, 1905). The bismuth was from several distinct sources, and included material received from Classen, whose determinations of this atomic weight were published several years ago.

In the first part of the research, bismuth was oxidised by means of nitric acid in porcelain crucibles. The oxide was tested for occluded gases, which were proved to be absent, or at least present in only insignificant traces. Three series of determinations are given, representing different samples of bismuth, with the subjoined results.

Weight Bi.	Weight Bi ₂ O ₃ .	Atomic weight.
9'63289	10'74328	208'22
10'41101	11'61288	208'04
10'97914	12'24528	208'11
10'1199	11'2880	208'10
18'96770	21'15541	208'08
11'99601	13'38001	208'01
27'23022	30'37392	207'88
24'9817	27'86431	207'99
10'11284	11'27998	207'94
28'35991	31'63053	208'10
Mean		208'05

By reduction of bismuth trioxide in a stream of ammonia gas, with precautions which are fully described in the original memoir, the following determinations were made.

Weight Bi ₂ O ₃ .	Weight Bi.	Atomic weight.
1'45827	1'30751	208'14
2'12432	1'90461	208'04
3'0021	2'6918	207'92
2'1012	1'8840	208'17
3'0182	2'70620	208'16
1'9091	1'71171	208'02
Mean		208'08

Birckenbach concludes, from all the determinations, that Bi = 208 approximately, and not 209 as measured by Classen.

The foregoing investigation was conducted under the direction of Gutbier, who has carried the work still further. In a preliminary notice, Gutbier states that eight analyses of bismuth bromide gave values for Bi ranging from 207'89 to 208'24, in mean 208'05 (*Zeit. Elektrochem.*, xi., 831). Additional determinations are being made.

Palladium.

In the elaborate memoir by Amberg (*Ann.*, cccxli., 235) on the atomic weight of palladium, several methods of determination are described; but all relate to analyses of palladosammine chloride, Pd(NH₃Cl)₂.

First, palladium was precipitated by electrolysis, and the chlorine in the remaining solution was weighed as AgCl. From the following data, the atomic weight A is determined by the proportion of Pd, B by the ratio between the original salt and AgCl.

Weight salt.	Weight Pd.	Weight AgCl.	A.	B.
1'06045	0'53609	—	107'38	—
1'00028	0'50528	1'35867	107'28	106'09
1'66386	0'84085	2'25437	107'33	106'59
0'83195	0'42092	1'12282	107'56	107'45
1'91591	0'96886	2'59799	107'44	106'45

In a second series of experiments the palladium was thrown down by hydrazine sulphate; in other respects this series is like the first.

Weight salt.	Per cent Pd.	Weight AgCl.	Atomic weight.
1'32423	50'12	1'78656	107'52
1'02642	50'29	1'39247	106'35
1'30335	50'36	1'76875	106'28
1'59709	—	2'16641	106'37
1'88622	50'49	2'55028	107'06
2'59665	50'68	3'51783	106'64

From the sum of all the chlorine determinations, Pd = 106'67 when Ag = 107'93, Cl = 35'45, N = 14'04, and H = 1'008. The weights are referred to a vacuum.

In the final series of determinations, palladium was electrolytically precipitated from a sulphuric acid solution of the palladosammine chloride with the aid of a rotating anode. Twelve analyses were made with three separate preparations of the chloride, and with the subjoined results:—

Weight salt.	Weight Pd.	Per cent Pd.	Atomic weight.
0'62446	0'31470	50'396	106'70
0'83878	0'42280	50'407	106'75
1'50282	0'75725	50'389	106'67
1'06704	0'53753	50'385	106'66
1'98342	0'99971	50'403	106'74
1'53093	0'77153	50'396	106'71
1'18995	0'59971	50'398	106'71
0'62635	0'31572	50'406	106'75
1'76110	0'88739	50'388	106'67
3'79639	1'91298	50'389	106'68
3'97553	2'00333	50'392	106'69
4'62100	2'32834	50'386	106'66

Sum 23'51777 11'85109 Mean .. 106'699

From the sums of the weights, Pd = 106'688. The probable value, derived from all the data, is put by Amberg at 106'7. If re-calculated with the new values for N and Cl, 14'01 and 35'473, the figure for palladium will be changed very slightly. The changes due to N and Cl respectively are in opposite directions, and nearly compensate each other.

Thorium.

R. J. Meyer and A. Gumpertz (*Ber.*, xxxviii., 817) have investigated the supposed complexity of thorium, and have failed to confirm the results announced by Baskerville. First, about 700 grms. of thorium nitrate were separated into seven fractions by precipitation with potassium

chromate. The fractions were converted into the octohydrated sulphate, which was then applied to the atomic weight determinations. It was weighed first as hydrate, then as anhydrous salt after heating to 400°, and finally ignited to oxide. The data thus obtained are as follows:—

Fraction.	Wt. hydrate.	Wt. Th(SO ₄) ₂ .	Wt. ThO ₂ .	At. wt.
I.	1·2463	0·9301	0·5793	232·4
I.	1·3261	0·9927	0·6184	232·5
IV.	1·3910	1·0344	0·6442	232·3
IV.	1·2543	0·9349	0·5821	232·2
VII.	0·8934	0·6680	0·4160	232·3
VII.	—	0·4296	0·2676	232·5

Secondly, thorium tetrachloride was fractionally sublimed in a stream of chlorine, and divided into three portions, namely—(1) the most volatile portion, "berzelium"; (2) the medium portion, "new thorium"; (3) the unsublimed residue, "carolinium." These, converted into sulphate, gave the subjoined data.

Fraction.	Wt. hydrate.	Wt. Th(SO ₄) ₂ .	Wt. ThO ₂ .	At. wt.
I.	1·2573	0·9199	0·5730	232·5
I.	1·0424	0·7647	0·4764	232·6
2.	—	1·0650	0·6635	232·4
2.	1·0387	0·7758	0·4834	232·7
3.	1·1904	0·8824	0·5496	232·4
3.	0·7487	0·5545	0·3454	232·4

The mean of all is Th=232·43. No evidence of a breaking up of thorium was observed, and a spectroscopic examination of the products by Eberhard led to the same conclusion (*Ber.*, xxxviii., 826). A brief reply to Meyer and Gumpertz was published by Baskerville (*Ber.*, xxxviii., 1444), in which he urged the importance of observing the exact conditions of his experiments—conditions from which his critics seem to have departed.

The radio-thorium described by Ramsay (*Journ. Chim. Phys.*, iii., 617) and Hahn (*CHEMICAL NEWS*, xci., 193; xcii., 251; *Ber.*, xxxviii., 3371; see also Sackur, *Ber.*, xxxviii., 1756) should be taken into account in any discussion of the atomic weight of thorium. It was extracted in very small amount from thorianite, and gave about 700,000 times as much emanation as an equal weight of ordinary thorium. Its atomic weight remains to be determined.

(To be continued).

A SYSTEM OF QUALITATIVE ANALYSIS INCLUDING NEARLY ALL THE METALLIC ELEMENTS.*

PART II.—ANALYSIS OF THE TUNGSTEN GROUP.

By ARTHUR A. NOYES.

(Continued from p. 192).

Procedure and Notes (continued).

Procedure 39.—Combine the filtrate from the (NH₄)₂S precipitate (P. 35) with that from the Na₂CO₃ and S fusion (P. 38). Make the solution distinctly acid with H₂SO₄ (1·20), shake, and filter, using suction if the precipitate is large. (Precipitate, if its colour is that of precipitated sulphur, reject; if not, P. 41. Filtrate, if a sulphide has been precipitated from it or if it is at all yellow, or if it contains phosphate, P. 40; otherwise, reject).

Notes.

1. Molybdenum, tin, and tungsten, when present in the H₂SO₄ precipitate, even in quantity as small as can be detected by the subsequent tests for these elements, impart either a dark colour or a deep yellow colour to that precipitate. Therefore, if the precipitate has the colour of pure sulphur, it is not necessary to examine it

further. In a doubtful case, the precipitate should be ignited as described in P. 41, and the amount of the residue estimated or weighed. Molybdenum is completely or nearly completely precipitated by the H₂SO₄, and tin is completely precipitated by it. Tungsten, when present in large quantity, divides itself between the precipitate and filtrate; when present alone in small quantity it passes almost wholly into the filtrate, to which even a quantity of 1 m.grm. imparts a distinct yellow colour. In the presence of the other elements precipitated as sulphides, a small quantity of tungsten may, according to the conditions, either be completely precipitated with them, or it may pass entirely into the filtrate, and without imparting to it a yellow colour. If a precipitate of sulphide is obtained, it is therefore necessary to test the filtrate for tungsten by P. 40, even though it be colourless. In the presence of phosphate, too, even a considerable quantity of tungsten seems to pass completely into the filtrate.

Procedure 40.—If the filtrate from the H₂SO₄ precipitate (P. 39) requires examination (see P. 39), evaporate it till the residue becomes almost dry, transfer the latter to a platinum crucible, and ignite as long as H₂SO₄ fumes are expelled. Add to the residue a little HF and a few drops H₂SO₄, and again evaporate till fuming ceases. Add to the residue twice its volume of solid Na₂CO₃, and heat for five minutes over a powerful burner. Cool, fill the crucible with water, heat till the mass is dissolved, and filter if the solution is not clear. Acidify the solution strongly with HNO₃, and boil. If no precipitate separates, evaporate the liquid to dryness, moisten with HNO₃ (1·20), again evaporate to dryness, warm the residue with enough HNO₃ (1·10) to dissolve the salt present, and allow time for any precipitate to settle out. Whether a precipitate forms or not, add 2 c.c. H₂SO₄ (1·84), evaporate to the fuming-point, dilute with 5 c.c. HCl (1·02), and add a little granulated Zn. (Residue and solution, reject).

Notes.

1. The treatment with HF and H₂SO₄ serves to remove any SiO₂ that may have been introduced by the action of the alkaline solutions on the vessels or in the fusion with Na₂CO₃ and S. If not removed, it would separate with the H₂WO₄ when the aqueous solution of the fused mass is evaporated with HNO₃, and might obscure the test for tungsten.

2. When HNO₃ is added to an alkaline tungstate, H₂WO₄ separates as a white, flocculent precipitate, which upon boiling turns yellow, and becomes more compact. A very small quantity of tungsten, however, may not precipitate in this way; but it is separated as a heavy yellow powder, or as yellow flocks, or as a yellow stain on the sides of the dish, when the solution is repeatedly evaporated with acid.

3. In case a distinct yellow precipitate separates from the HNO₃ solution, the evaporation with H₂SO₄ and the addition of Zn, whereby a deep blue colouration and precipitate are produced if tungsten is present, serves only as an (almost superfluous) confirmation of the presence of that element. If, however, phosphate is present, and there is no yellow precipitate, the reduction test with Zn is essential, since H₂WO₄ does not separate from a solution containing a corresponding amount of this acid radical. Even a large quantity of phosphate does not, however, prevent the production of a strong blue colour with 1 m.grm. of tungsten.

Procedure 41.—If it is large enough in quantity, separate the H₂SO₄ precipitate (P. 39) from the filter, and transfer it to a porcelain crucible. If small in quantity, pour a portion of 2–3 c.c. (NH₄)₂S repeatedly through the filter till the precipitate is dissolved, collect the solution in a porcelain crucible, and evaporate it to dryness. Provide the crucible with a perforated porcelain cover through which passes a porcelain tube. Pass through the tube into

* From the *Technology Quarterly*, xvii., No. 3.

the crucible a slow current of H_2S gas, and heat the crucible (preferably within a cylindrical jacket with open ends, if the precipitate is large) first gently, then more strongly, and finally for five to thirty minutes, or until the whole mass becomes black, to the highest temperature attainable with a powerful burner. Allow the crucible to cool with the H_2S gas still passing through it. Rub its contents to a powder, and transfer them to a small Erlenmeyer flask. Pour into the crucible, if some of the substance is adhering to it, 2–3 c.c. HCl (1·20), warm, and pour the solution, together with 5–10 c.c. more HCl (1·20), into the flask. Cover the flask, and boil the liquid gently as long as the vapours continue to blacken filter-paper wet with a NaOH solution of $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$, replacing the acid which evaporates. Dilute the solution with its own volume of water, filter, and wash the residue. (Residue, P. 43; solution, P. 42).

Note.

1. Precipitated WS_3 and V_2S_5 are sufficiently soluble in hot strong HCl to make their separation from SnS_2 and Sb_2S_3 by this solvent impracticable. By ignition in an atmosphere of H_2S , however, they are converted into WS_2 and VS_2 , which are entirely insoluble in boiling HCl (1·20). The ignition also serves to volatilise any As_2S_3 present, which otherwise would prevent the precipitation of H_2WO_4 in the subsequent test for tungsten. By the ignition SnS_2 and Sb_2S_3 are converted into black SnS and Sb_2S_3 respectively, which dissolves readily in HCl (1·20). If the ignition is not prolonged or intense enough, the hydrated SnS_2 is converted only into "mosaic gold" (SnS_2), consisting of golden yellow scales with metallic lustre; and this is only very slowly dissolved by the hot acid. The ignition must therefore be continued till the entire mass becomes black. Both MoS_3 and MoS_2 into which it is converted by ignition are entirely unattacked by boiling hydrochloric acid. The H_2S prevents the conversion of the sulphides into oxides, which occurs if air has access to them at a high temperature.

Procedure 42.—Evaporate the HCl solution (P. 28) to a volume of 2–3 c.c., and add an equal volume of water and about 0·5 grm. finely granulated Zn . Allow the action to continue for ten minutes. Decant the solution completely from the residue, allow any suspended particles to settle out from the liquid, and decant again. (Solution, reject). Add to the united residues 2–4 c.c. HCl (1·20), heat gently till the Zn has all dissolved, and then just to boiling unless the residue has previously disappeared, add half a volume of water, and pour the solution through a small filter into 5 c.c. nearly saturated HgCl_2 solution. (Residue, precipitate, and solution, reject).

Notes.

1. If tin seems to be present in considerable quantity (10 m.grms. or more), it may advantageously be tested for more simply by adding a portion of the HCl solution of the ignited sulphide, immediately after the H_2S is completely boiled out, to HgCl_2 solution; and in case a precipitate results, the treatment with Zn may be dispensed with; for after the ignition the tin is in the stannous state, and no other element can be present which reduces HgCl_2 , since ignited WS_2 , MoS_2 , and VS_2 are entirely insoluble in HCl , and since SbCl_3 does not cause reduction. In case, however, only a small quantity of tin (5 m.grms. or less) is present, it may become completely oxidised during the boiling of the HCl solution. In order that the tin test may be delicate, it is therefore necessary to follow the procedure and observe the precautions above indicated—especially to allow time for the complete reduction of the tin salt by the Zn , to secure solution of the tin by heating sufficiently with strong HCl , and to prevent oxidation of the stannous salt by carrying out rapidly the last steps in the process. By observing these precautions 1 m.grm. of tin is readily detected.

2. It is not necessary to test for antimony at this point, since it would have been precipitated in large part by H_2S in the first procedure of this group, and would pass, partly through that precipitation, and partly directly into the main HNO_3 solution.

Procedure 43.—Heat the residue insoluble in HCl (P. 41) in a covered vessel on a steam-bath with 2–8 c.c. HNO_3 (1·42) and 1–2 c.c. HCl (1·20) until it all dissolves, with the exception of a few black particles, or becomes pure yellow. If there is a residue, add an equal volume of water, allow the residue to settle, and decant the liquid through a filter. To the filtrate, or to the unfiltered solution if there was no residue, add 2–3 c.c. H_2SO_4 (1·84), evaporate till this acid fumes slightly, keep at that temperature for three or five minutes, cool, add 5 c.c. water, allow the residue to settle, and decant the solution through a filter. (Residues, P. 44; solution, P. 45).

Notes.

1. Even 100 m.grms. of ignited MoS_2 are completely dissolved by 10 c.c. of the mixture of HNO_3 and HCl ; and as a larger quantity cannot be present in this precipitate, there is no danger of its interfering with the tungsten test, provided the acid used is not evaporated off. If, however, the acid solution is allowed to concentrate, MoO_3 may separate as a white or light yellow powder, which will re-dissolve only in a very large quantity of HNO_3 . If this seems to have occurred, the residue should not be separated from the solution till after the evaporation with the H_2SO_4 . Black particles of carbon are usually left undissolved by the HNO_3 and HCl .

2. Even a small quantity (1–2 m.grms.) of tungsten, when present alone at this point, is readily detected by its separation as a yellow residue from the HNO_3 and HCl solution; but when much molybdenum (100 m.grms.) is also present, a considerable quantity of tungsten (at least 4 m.grms.) remains in solution. After the heating with the H_2SO_4 , however, a quantity even as small as 2 m.grms. always separates as a yellow compact powder.

3. If no yellow residue is obtained here or from the filtrate from the H_2SO_4 precipitate (P. 40) it does not show the absence of tungsten in the substance, since it may have passed entirely into the main HNO_3 solution (P. 6), as mentioned in P. 5, N. 1.

Procedure 44.—Add to the residues insoluble in HNO_3 and H_2SO_4 (P. 43) 2–3 c.c. HCl (1·12), evaporate to dryness, add 2 c.c. HCl (1·12) and a few granules of Zn .

Note.

1. The evaporation with HCl removes the HNO_3 adhering to the residue. Even in the presence of 0·5 m.grm. W , a distinct blue colouration results, while with a somewhat larger quantity the mixture assumes a dark blue colour of great intensity. This colour is due to a suspended precipitate, which soon coagulates, consisting of a hydrated oxide intermediate between WO_3 and WO_2 . Molybdenum when present in strong HCl solution, such as is directed to be used in this test, does not give a blue colour with Zn , but there results a series of colours—yellow, dark orange, dark green, and, finally, dark olive-brown. In dilute HCl solution, however, molybdenum, even in moderate quantity, gives rise at once to a deep blue colouration of the solution.

Procedure 45.—Add to the H_2SO_4 solution (P. 43) of the ignited sulphides 1–2 c.c. 10 per cent KSCN solution and some granulated Zn .

Note.

1. A red colouration which appears on the addition of the Zn shows the presence of molybdenum. Iron, if present as impurity, will give a red colour with the

KSCN alone, but this disappears almost immediately on the addition of the Zn. A negative result is of no significance, since molybdenum may have passed entirely into the main HNO₃ solution.

(To be continued).

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, April 5th, 1906.

Prof. R. MELDOLA, F.R.S., President, in the Chair.

(Concluded from p. 198).

76. "The Dynamic Isomerism of Phloroglucinol." By EDGAR PERCY HEDLEY.

It is well known that alkalis and acids affect the equilibrium existing between two isomerides in solution, and that the action of the alkali is to increase the oscillatory change in one direction, whilst the acid, on the other hand, decreases it.

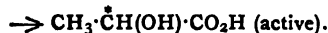
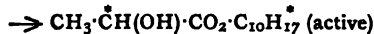
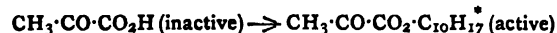
If phloroglucinol exists in solution in two forms—the enolic and ketonic modifications—then the position and persistency of the absorption band ought to undergo modification in the presence of alkali, and ought, in fact, to be displaced towards the less refrangible end of the spectrum. On the other hand, in the presence of acid, the persistency of the band ought to be decreased and become more like that of phloroglucinol trimethyl ether. These arguments were fully borne out by experimental test.

Lowry has pointed out that in some cases the solvent affects the equilibrium between dynamic isomerides. It was therefore thought advisable to examine whether in the case of phloroglucinol the solvent had any effect. Any non-ionising solvent would be suitable, provided it transmitted the ultra-violet light to a sufficient extent. Accordingly ether, a good and diastinct solvent, was used, and was found to have no effect on the spectrum.

The following conclusions were established:—(1) That in neutral solutions phloroglucinol exists in both modifications, the enol being greatly in preponderance over the keto-form; and (2) that this equilibrium is undisturbed by the class of solvent.

77. "Studies in Asymmetric Synthesis. V. Asymmetric Syntheses from *l*-Bornyl Pyruvate." By ALEXANDER MCKENZIE and HENRY WREN.

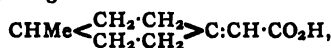
The authors have examined the reduction of *l*-bornyl pyruvate by aluminium amalgam, and find that an asymmetric synthesis of *l*-lactic acid may be accomplished in accordance with the scheme:—



The actions of magnesium ethyl iodide, magnesium isobutyl iodide, magnesium phenyl bromide, and magnesium α -naphthyl bromide respectively on *l*-bornyl pyruvate, and of magnesium isobutyl iodide and magnesium α -naphthyl bromide respectively on *l*-menthyl pyruvate, have been studied. The results obtained are contrasted with those recorded in a previous paper with the object of comparing the effect, first, of the active menthyl and bornyl groups, and, secondly, of the various alkyl (or aryl) halides used on the extent of the asymmetric syntheses of the resulting substituted glycolic acids.

78. "*l*-Methylcyclohexylidene-(4)-acetic Acid." By WILLIAM HENRY PERKIN, jun., and WILLIAM JACKSON POPP.

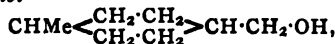
The current number of the *Berichte* (1906, xxxix., 1171) contains a paper by W. Marckwald and R. Meth, in which these investigators describe the synthesis of an acid to which they assign the formula—



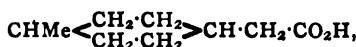
and which they have resolved into active modifications by fractional crystallisation of the cinchonine salts.

During the last three years, the authors have been engaged in an attempt to prepare a substance which, while not containing an asymmetric carbon atom is yet capable of existing in optically active modifications, and it is noteworthy that one of the substances selected for examination was methylcyclohexylideneacetic acid. Since, however, the method of preparation is quite different from that of Marckwald and Meth, and the results obtained are not in agreement with theirs, the following preliminary account of the authors' experiments is now put forward.

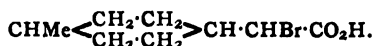
Ethyl hexahydro-*p*-toluate is converted into hexahydro-*p*-tolylcarbinol—



by reduction with sodium and alcohol. This carbinol distils at 197°, and is readily converted into hexahydro-*p*-tolyl bromide (b. p. 135°/150 m.m.) by the action of hydrobromic acid at 120°. When this bromide is heated with potassium cyanide and the product hydrolysed, it yields hexahydro-*p*-tolylacetic acid,—



which melts at 73–74°, and is readily acted on by phosphorus pentachloride and bromine with formation of α -bromohexahydro-*p*-tolylacetic acid (m. p. 78°),—



The ester of this bromo-acid is decomposed by boiling with diethylaniline with elimination of hydrogen bromide and formation of ethyl methylcyclohexylideneacetate, which distils at 158°/100 m.m., and, on hydrolysis, yields the free acid,—

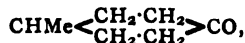


The methylcyclohexylideneacetic acid thus obtained crystallises from formic acid in glistening plates, and melts at 70–71°, whereas the acid described by Marckwald and Meth, and to which these authors assign this constitution, melts sharply at 40–41°.

The method of preparation seems to leave no doubt that the acid melting at 70–71° is methylcyclohexylideneacetic acid, whilst the acid of melting-point 40–41° described by Marckwald and Meth is possibly the isomeric acid,—



The fact that, when heated with strong caustic potash, it is decomposed into 4-methylcyclohexanone,—



and acetic acid is easily explained on this assumption.

It is well known that an unsaturated acid which contains the double linking in the $\beta\gamma$ -position is readily converted, by the action of strong caustic potash, into the corresponding $\alpha\beta$ -unsaturated acid, and this isomeric change may have taken place in the case of Marckwald and Meth's acid prior to the decomposition into methylcyclohexanone and acetic acid.

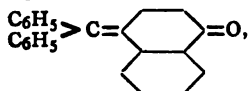
The authors are investigating this matter further, and are also engaged in a series of experiments with the object of resolving the acid melting at 70–71° into optically active modifications.

79. "Condensation of Benzophenone Chloride with α - and β -Naphthols." By GEORGE WILLIAM CLOUGH.

By heating α -naphthol with benzophenone chloride, the author has prepared di- α -hydroxynaphthylidiphenylmethane, the acetyl and benzoyl derivatives of which have also been obtained.

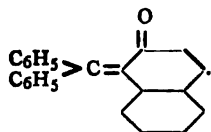
When benzophenone chloride was added to a boiling solution of β -naphthol in xylene, the product obtained was di- β -naphthoxydiphenylmethane, which is hydrolysed by dilute sulphuric acid into β -naphthol and benzophenone.

The action of sodium α -naphthoxide on benzophenone chloride resulted in the formation of the inner anhydride of α -naphthylidiphenylcarbinol, and the same substance was also obtained by adding α -naphthol to a boiling solution of the ketone chloride in light petroleum. This compound is yellow, and the quinonoid formula,—



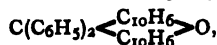
has been assigned to it.

Sodium β -naphthoxide interacts with benzophenone chloride in a similar manner to form a red, crystalline substance, to which the following formula is ascribed:—



These coloured compounds are soluble in concentrated sulphuric, nitric, and hydrochloric acids respectively, the α -compound giving violet and the β -compound green solutions in these acids.

A condensation of benzophenone itself with α -naphthol has also been effected by heating these substances in the presence of zinc chloride and hydrogen chloride at 150–160°. The product obtained was α -oxydinaphthylidiphenylmethane,

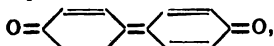


the condensation resembling that of acetone with α -naphthol (Dianin, Abstr., 1893, i., 214). Its solution in concentrated acid is yellow and exhibits a green fluorescence.

80. "The Constitution of Cœrulignone (Cediret)." (A Preliminary Note). By JAMES MOIR.

On the authority of Liebermann, Hofmann, and others, it has been accepted that cœrulignone is tetramethoxydiphenoquinone; in addition, several strictly analogous substances have been prepared and formulated by their discoverers as the corresponding tetra-chloro-, -bromo-, -amino-, and -hydroxy-derivatives of diphenoquinone.

Now diphenoquinone itself,—



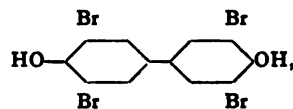
has recently been prepared by Willstätter and Kalb (Ber., 1905, xxxviii., 1232), and the author finds that its properties are quite different from those of cediret and its analogues, and that, in fact, diphenoquinone is a true para-quinone, whereas cediret and its analogues do not possess any quinonoid property except colour. Their properties are:—(1) Complete insolubility in organic solvents; (2) metalloid lustre; (3) decomposition on heating without melting or subliming; (4) intense colourations—blue to crimson—with sulphuric acid; (5) characteristic colour reactions with alkali; (6) production of complex sulphonates of high molecular weight through the action of sodium sulphite. Such properties as these indicate molecular complexity and a polyquinonoid constitution.

The author finds that the "diphenoquinhydrone" of Willstätter and Kalb (*loc. cit.*) is the true parent substance of cediret, since it possesses all the foregoing properties. Nevertheless, it is doubtful at present whether this sub-

stance is really a simple quinhydrone; that is, a direct additive product of diphenoquinone and diphenol; some of its reactions indicate a larger molecular weight and higher percentage of oxygen than the formula $\text{C}_{24}\text{H}_{18}\text{O}_4$ requires.

The substance described by Magatti as tetrabromodiphenoquinone (Ber., 1881, xiii., 226) has been studied more in detail. In the first place, it is quite different from the true tetrabromodiphenoquinone, which the author has prepared by oxidising tetrabromodiphenol with lead peroxide, and which forms transparent orange crystals, is soluble in many organic solvents, and otherwise resembles diphenoquinone. Magatti's substance contains a lower percentage of bromine than is required by such a formula as $\text{C}_{12}\text{H}_4\text{O}_2\text{Br}_4$, and Magatti's analysis in support of this formula may be set aside as probably fictitious, since he has miscalculated the theoretical proportion of bromine and yet his analysis agrees with the assumed theoretical value. The compound gives, even in traces, a magnificent crimson shade with sulphuric acid, and on digestion with alkali forms a derivative which closely resembles indigotine in colour and coppery lustre; this can be also prepared directly by careful oxidation of an alkaline solution of tetrabromodiphenol with iodine or potassium ferricyanide.

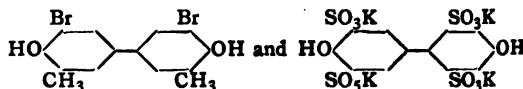
The orientation of the bromine atoms in tetrabromodiphenol has been settled to be as in the formula—



(3 : 5 : 3' : 5'-tetrabromo-4 : 4'-diphenol).

since the sole product of the oxidation of its diacetate was 3 : 5-dibromo-4-acetylbenzoic acid.

Finally, two new analogues of cediret have been prepared by oxidising the compounds—



respectively; the former has a green, graphite-like lustre, and gives a maroon shade with sulphuric acid, whilst the latter, easily soluble in water, has a very dark indigo colour.

81. "A Comparative Crystallographic Study of the Perchlorates and Permanganates of the Alkalies and the Ammonium Radicle." By THOMAS VIPON BARKER.

The perchlorates of potassium, rubidium, caesium, and ammonium form an isomorphous group similar to that of the permanganates of the same metals, which were investigated by Muthmann. The salts are orthorhombic, and possess perfect cleavages parallel to the basal plane and the prism. All the constants of rubidium perchlorate determined are intermediate between those of potassium and caesium perchlorates; ammonium perchlorate, again, lies very close to rubidium perchlorate, as does also thallium perchlorate according to Roscoe's measurements. The series, therefore, resembles the isomorphous groups investigated by Tutton. On comparing the perchlorates with the permanganates, it is found that the effect of replacing an atom of chlorine by one of manganese is much the same as that induced by the substitution of sulphur by selenium, say, in the sulphates of the same metals. The crystallographic evidence for placing manganese in the seventh group of the periodic classification, so far as such evidence goes, is therefore of the strongest possible kind.

Five crops of isomorphous mixtures of potassium perchlorate and permanganate of varying composition were measured. In one crop only, corresponding very closely to the formula $\text{K}_2(\text{ClO}_4)(\text{MnO}_4)$, were abnormal angles found; that is, angles the values of which lie outside those of the constituent salts; moreover, these angles were all in one zone, in which the faces were possibly vicinal.

82. "Contributions to the Theory of Isomorphism based on Experiments on the Regular Growths of Crystals of one Substance upon those of Another." By THOMAS VIPOND BARKER.

The parallel growths of sodium nitrate on cleavage surfaces of calcite are independent of the locality or variety of the calcite so long as a good fresh cleavage surface is obtainable. Regular growths of rhombohedra of the nitrate were also obtained upon certain other forms of calcite, for example, the prism, scalenohedron, a steep rhombohedron, and the rare prism $\{110\}$; in all cases a pair of similar edges of the calcite and sodium nitrate are congruent. The effect of a magnetic field on the orientation of the crystals was purely negative. No parallel or regular growths could be obtained on the other minerals of the calcite group—dolomite, calamine, chalybite, rhodocroite, diallogite, and breunnerite—nor on barytocalcite. The failure of the latter minerals to induce a parallel deposition is probably not due to a difference of symmetry or of angle (or axial ratio), but to a dissimilarity of molecular volume, and hence of topic axes also.

This view is strengthened by the discovery of parallel growths among the members of another and more numerous series of isostructural minerals and salts; potassium perchlorate and permanganate are deposited (from aqueous solution) upon barytes, celestine, and anglesite in parallel position, whereas the perchlorates of rubidium, caesium, ammonium, and thallium and the permanganates of rubidium, caesium, and ammonium are not. Here, again, those salts which do form parallel growths are characterised by a closeness of molecular volume to those of the minerals on which they are deposited, while similarity of angle (or of axial ratios) is not sufficient to induce a parallel growth. If the above substances be arranged in order of topic axes, it is seen that potassium perchlorate and permanganate fall next to the three minerals mentioned.

Since calcite and sodium nitrate resemble each other so closely in all their physical properties, and since calcite relieves supersaturation in a metastable solution of the nitrate, the conclusion is drawn that these isostructural substances are also to be regarded as isomorphous, although mixed crystals are not obtainable.

Many unsuccessful attempts were made to obtain regular growths of other salts on other minerals, more especially of cubic salts on cubic minerals and potassium nitrate on aragonite; but regular growths of potassium bromide, iodide, and nitrate and sodium nitrate on mica, and of quinol on calcite, were obtained.

All the perchlorates and permanganates mentioned yield parallel growths on each other, but it was found that pairs of isomorphous salts, the molecular volumes of which are almost identical, form zonal growths ("Schichtkrystalle") rather than parallel growths.

83. "Constitution of Silicin. Synthesis of Pentamethyl Salicin." By JAMES COLQUHOUN IRVINE and ROBERT EVSTAFIEFF ROSE.

Salicin was alkylated by means of the joint action of silver oxide and methyl iodide, with the production of pentamethyl salicin. The process was carried out in the first instance in methyl-alcoholic solution, and, after the alkylation had proceeded far enough, methyl iodide was used as solvent. The product crystallised in delicate needles (m. p. 62–64°), readily soluble in organic solvents and giving $[\alpha]_D^{20} = 52.15^\circ$ in alcoholic solution.

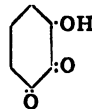
Emulsion was found to be without action on the compound, and even dilute mineral acids readily converted it into a resinous mass resembling saliretin. The hydrolysis was therefore carried out indirectly by prolonged heating at 100° of a solution of the substance in methyl alcohol containing 0.25 per cent of hydrochloric acid. Tetramethyl glucose was thus produced, and afterwards converted into the equilibrium mixture of α - and β -tetramethyl methylglucosides. The alkylated glucosides were isolated and then hydrolysed to give tetramethyl glucose (m. p. 84–86°).

This reaction proves that salicin (and hence also helicin and populin) contains the same γ -oxidic linking as the methylglucosides and sucrose.

Further evidence on this point was obtained in the synthetical preparation of pentamethyl salicin. On heating saligenin and tetramethyl glucose to 120° in benzene containing 0.25 per cent of hydrochloric acid, condensation took place with the formation of a mixture of saligenin tetramethyl glucosides and octamethyl glucosidoglucoside. After separation of the alkylated dissaccharide, the mixture was methylated by the silver oxide reaction and a compound, identical in every respect with the pentamethyl salicin prepared directly from the glucoside, was isolated from the product.

84. "A Product of the Action of Isoamyl Nitrite on Pyrogallol." By ARTHUR GEORGE PERKIN and ALEC BOWRING STEVEN.

When an alcoholic solution of pyrogallol is oxidised with acetic acid and isoamyl nitrite, a small quantity (1.3 per cent) of pale salmon-coloured leaflets separate (m. p. 203°). The main bulk of this product is colourless, may be obtained as minute prisms melting at 206–208° with effervescence, and has the composition $C_6H_4O_3$. With acetic acid and zinc dust it gives pyrogallol with acetic anhydride and zinc dust triacetylpyrogallol (m. p. 160–162°); its acetyl derivative, $C_6H_3O_3(C_2H_5O)$, forms colourless leaflets (m. p. 283–285°), and also yields triacetylpyrogallol in a similar manner. The substance $C_6H_4O_3$ dissolves sparingly in boiling acetic acid and alcohol with some decomposition, and by means of boiling water yields purpurogallin, $C_{12}H_8O_5$. The reaction studied in detail with the crude substance (m. p. 203°) showed that in this way a compound $C_{12}H_{10}O_7$? (colourless prisms, m. p. 242–243°) and an orange-brown resin, both readily soluble in water, are also formed. Owing to the poor yield it has not been possible to study this product further, but it appears likely that it may consist of hydroxy-o-benzoquinone:—



85. "A Reaction of Ellagic and Flavellagic Acids." By ARTHUR GEORGE PERKIN.

When ellagic acid is heated at about 230° with 100 per cent sulphuric acid, it is oxidised with formation of a new compound which crystallises from pyridine in small, yellow, prismatic needles and is soluble in strong alkalis with a greenish yellow tint, changing to blue on dilution. It appears to have the formula $C_{14}H_6O_{10}$ (found, C = 50.01; H = 2.17), gives a hexacetyl derivative [needles, $C_{14}H_{10}(C_2H_5O)_6$, m. p. 324–329°; found, C = 53.18; H = 3.32; $C_{14}H_6O_{10} = 57.24$], and dyes mordanted fabrics more readily than ellagic acid. Flavellagic acid behaves similarly [found for $C_{14}H_6O_{10}$, C = 50.09; 2.08; and for $C_{14}H_{10}(C_2H_5O)_6$, C = 53.12; H = 3.34 per cent].

86. "Some Thio- and Dithio-carbamide Derivatives of Ethylenaniline and the Ethylenetoluidines." By OLIVER CHARLES MINTY DAVIS.

In the interaction of ethylenaniline and the ethylenetoluidines with the thiocarbimides, several instances of steric hindrance were observed, the position of the substituent methyl group in the ethylenetoluidines and the thiolthiocarbimide having an important bearing both on the time of reaction and also on the nature of the product.

One molecule of ethylenaniline unites with two molecules of allyl-, phenyl-, o-tolyl-, or p-tolyl-thiocarbimide to form symmetrical dithiocarbamide derivatives, but with m-tolylthiocarbimide only one molecule of each unites to form an asymmetrical substituted thiocarbamide. Ethylene-toluidine reacts with difficulty to give asymmetrical derivatives with phenyl-, m-tolyl-, and p-tolyl-thiocarbimides, whilst with allyl- and o-tolyl-thiocarbimides the

yield was exceedingly small. Ethylene-*m*-toluidine gives symmetrical derivatives with phenyl- and *p*-tolylthiocarbimides, but with allyl-, *o*-tolyl-, and *m*-tolylthiocarbimides asymmetrical derivatives result. Ethylene-*p*-toluidine gives an asymmetrical derivative with *m*-tolylthiocarbimide, but with other thiocarbimides symmetrical derivatives are formed.

With the ethylenetoluidines, the ortho- and meta-positions therefore seem to modify the reactions, but with the thiocarbimides the meta-position only has this effect.

The foregoing compounds interact with mercuric oxide in alcoholic solution when heated at about 140° in sealed tubes, oxygen replacing sulphur. The product from ethylenedianiline and phenylthiocarbimide appears to be identical with a compound synthesised from ethylenedianiline and phenylcarbimide.

OBITUARY.

PROF. PIERRE CURIE.

ONE of the saddest deaths it has been our painful duty to record is that of the late Prof. Pierre Curie, who, with his wife, was the discoverer of the element radium. He met his death by being knocked down by a waggon while crossing the Rue Dauphine, Paris, on April 19th; his head was crushed, and death must have been instantaneous.

By his death, which has occurred in the flower of his manhood, we lose a great personal friend, and the world loses one of its most brilliant scientific men. We well remember our first visit to his laboratory some years ago, when much less was known of radium than is now. It was as modest and unassuming in appearance as was the great discoverer himself; it was, in fact, little better than a shed almost hidden by the surrounding buildings; but in this shed was first brought to light an element which, though even now known to exist in only the most minute proportions in any known mineral, has already revolutionised the whole train of scientific thought with regard to the construction of the atom and the conservation of matter as we know it. It is needless in these pages to do more than thus lightly pass over the properties of radium—they are so well known to all our readers. We have here to do more with the man himself than the details of his work.

Prof. Curie was born in Paris in the year 1859, and was therefore forty-six years of age; he obtained practically all his education in free classes under his father's guidance and as assistant in chemical laboratories, and finally graduated at the Sorbonne, where he attained the degree of Doctor of Science. His first researches were on the subject of piezo-electricity, in conjunction with his brother, and the skill and experience he attained in this work in the construction and manipulation of electrometers, guard-ring condensers, aperiodic balances, &c., must have been of the greatest service to him in his later more important work carried out in conjunction with his wife.

In 1895 Prof. Curie married Marie Sklodowska, one of the senior students at the Municipal School at which he was Professor, and it was through the combined efforts of these two highly-gifted students of Nature that the wonderful results were obtained that so startled the scientific and popular world a few years ago.

In 1903 M. and Mme. Curie received the Davy Medal of the Royal Society, and they shared with M. Branly the Osiris Prize and with M. Becquerel the Nobel Prize for physics.

Like many other great discoveries, theirs was received at first with indifference—even so recently as in the 1900 Exhibition in Paris, where it was practically overlooked; then with scepticism, until Mme. Curie published her

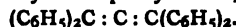
famous Thesis which gained for her the degree of Doctor of Physical Science. After this time honours and prizes were showered upon them, and the name of Curie obtained an imperishable place on the scroll of fame.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

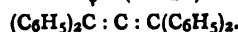
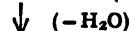
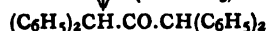
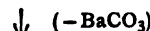
NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Berichte der Deutschen Chemischen Gesellschaft.
Vol. xxxix., No. 5, 1906.

New Aromatic Hydrocarbons.—D. Vorländer and C. Siebert.—By distilling barium diphenyl acetate a new crystalline colourless hydrocarbon is obtained; this the authors find to be sym-tetraphenyl allene,—



Apparently a part of the salt yields tetraphenyl acetone, and this is decomposed to form tetraphenyl allene and water on distillation.

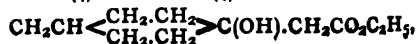


The water converts another part of the barium diphenyl acetate into diphenyl methane. The new substance is unsaturated, melts at 164°, and gives very unstable addition products with sulphuric acid and halogen hydrides. When treated with hydriodic acid and phosphorus or sodium and alcohol, it takes up hydrogen and yields a saturated hydrocarbon tetraphenyl propane, $(C_6H_5)_2CH.CH_2.CH(C_6H_5)_2$ (m. p. 140°). When oxidised with chromic acid it yields benzophenone and carbon dioxide. It is very readily attacked by acids and an isomeric hydrocarbon (m. p. 135°) is formed when it is heated with acetic acid or aqueous hydrochloric acid, or when it is allowed to stand with concentrated sulphuric acid or halogen hydrides in glacial acetic acid. This isomeric substance is also unsaturated, but yields no benzophenone with chromic acid.

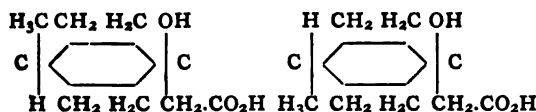
Combustion of Cadmium.—W. Manchot.—It is generally assumed that the brown fumes which are formed when cadmium is burnt consist of cadmium oxide, but it may very easily be shown that they contain cadmium peroxide also. If the fused metal is ignited and the fumes are collected in a beaker, and acidified potassium iodide added, a blue coloration appears. It is essential that the combustion should take place at as low a temperature as possible; if the metal is coated with a layer of oxide the experiment fails because it has to be heated too strongly in order to produce the fumes. In this case it may be observed that a potassium iodide solution, which has already turned blue, is again decolourised, as metal distils with the oxide. In this respect cadmium is allied to the alkali metals and magnesium.

Optically Active Compounds which contain no Asymmetric Atom.—W. Marckwald and R. Meth.—Inosite has hitherto been the only compound known which is optically active and yet contains no asymmetric atom, but the authors have now succeeded in preparing another similar compound. Starting from 1-methyl-cyclohexanone—

(4), $CH_3.CH<\begin{smallmatrix} CH_2.CH_2 \\ CH_2.CH_2 \end{smallmatrix}>CO$, by the action of brom- or iodo-acetic ester and magnesium, they obtained 1-methyl-cyclohexanol-(4)-acetic ester-(4),—



from which a mixture of two crystalline acids could be obtained. These two acids have the formulæ:—



If the cinchonine salts of these acids are prepared in alcoholic solution a syrup is obtained which crystallises after it has stood for some time in the desiccator. When the crystals are washed with ligroin and the salt decomposed with sulphuric acid the *d*-acid is obtained, while the ligroin solution with which the cinchonine salt was washed contains the *l*-acid.

MEETINGS FOR THE WEEK.

- MONDAY, 7th**—Society of Arts, 8. (Cantor Lectures). "Ivory in Commerce and in the Arts," by Alfred Maskell.
Royal Institution, 5. General Monthly Meeting.
Society of Chemical Industry, 8. "Notes on the Gutzeit Test for Arsenic," by J. Goode and F. M. Perkins. "Separation of Brucine and Strychnine—Influence of Nitrous Acid in Oxidation by Nitric Acid," by W. C. Reynolds and R. Sutcliffe. "Absorption of Gallic Acid by Organic Colloids," by W. P. Dreaper and A. Wilson.
- TUESDAY, 8th**—Royal Institution, 5. "Glands and their Products," by Prof. William Stirling, M.D., &c.
Society of Arts, 8. "Damascening, and the Inlaying and Ornamenting of Metallic Surfaces," by Sherard Cowper-Coles.
- WEDNESDAY, 9th**—Society of Arts, 8. "Bridge Building by means of Caissons, including remarks upon Compressed Air Illness," by Prof. Thomas Oliver, M.D., &c.
- THURSDAY, 10th**—Royal Institution, 5. "The Expansion of Old Greek Literature by Recent Discoveries," by the Rev. J. P. Mahaffy, C.V.O., D.D., &c.
- FRIDAY, 11th**—Royal Institution, 9. "Some Astronomical Consequences of the Pressure of Light," by Prof. J. H. Poynting, F.R.S., &c.
Physical, 8. "Effect of Rapid Discharge on the Throw of a Galvanometer," by A. Russell. Exhibition of Lippmann Capillary Dynamo and Electromotor, by Prof. H. A. Wilson. Exhibition of an Apparatus for Demonstrating the Movements of the Diaphragms of Telephonic Transmitters and Receivers and the Current flowing into and out of the Cable during Speech, by W. Duddell.
- SATURDAY, 12th**—Royal Institution, 3. "English Furniture in the Eighteenth Century," by Prof. Charles Waldstein, Ph.D., &c.

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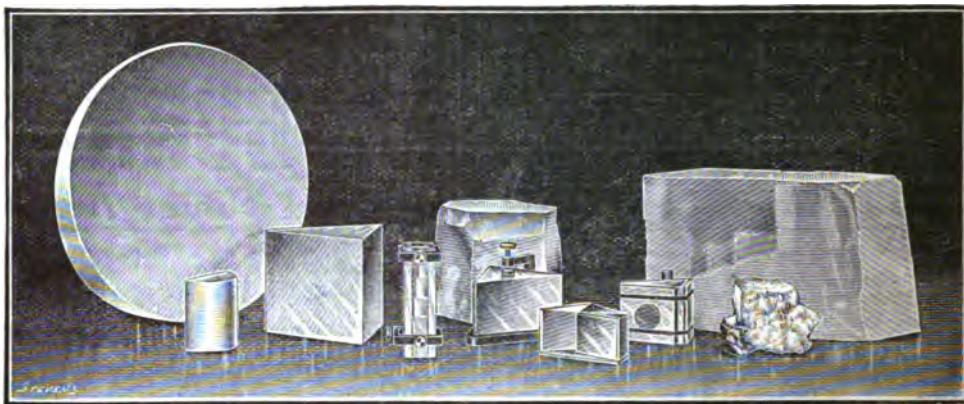
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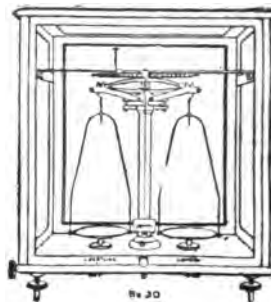
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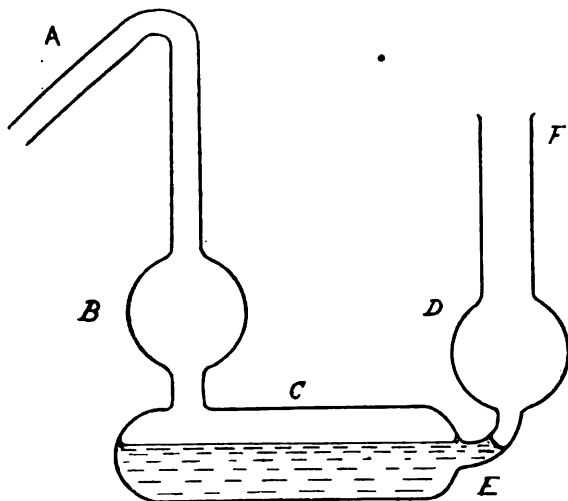
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VOL. XCIII., No. 2424.

A NEW FORM OF ABSORPTION-TUBE.

By EDGAR PHILIP PERMAN.

IN estimating chlorine (from peroxides, &c.), ammonia, and other substances in which an absorption-tube is used, difficulty is often experienced owing to the condensation of steam or the too rapid absorption of the gas evolved. The result is that some of the absorbing liquid is carried back into the flask in which the gas is generated. This difficulty can be entirely overcome by the use of a tube



of the pattern shown in the accompanying diagram. Convenient dimensions are C, 1 inch diameter, 3.5 inches long, B and D 1.25 inches diameter. The liquid should just close the narrow tube at E. A is connected with the generating flask by rubber, cork, or ground-glass joint, and a guard tube, containing glass beads moistened with the absorbent, may be connected to F. I have tested the tube as severely as possible, but it has never failed.

University College, Cardiff.

DETERMINATION OF SULPHUR IN PYRITES.

By C. R. GYZANDER,
Chemist to the Cochrane Chemical Co., Boston, U.S.A.

THOUGH there exists no method for determining what might reasonably be termed available sulphur in pyrites, from the sulphuric acid manufacturers point of view, Dr. Lunge's method aims at least towards this end; hence the reason for its almost universal adoption for the valuation of pyrites for the sulphuric acid trade.

But this method requires a good deal of manipulative skill, strict attention to details, is not well adapted for carrying on many simultaneous determinations, and is rather tedious. It is therefore much more suitable for the student than for the works chemist.

Of the many proposed improvements of this method none has yet proved of any particular value, either as regards final results or time required. But although the

time required for an analysis of this nature is secondary in importance, a method which materially shortens it without sacrificing accuracy is certainly to be preferred.

Years ago an improvement of the Lunge method was proposed, based on the reduction of the iron to the ferrous state by means of thiosulphate before precipitating with barium chloride.

Obviously the results, as regards percentage of sulphur, must be very incorrect by this method, but nevertheless it taught one important and valuable lesson, namely, that it was possible to precipitate BaSO_4 in the presence of iron, and free from iron when the latter is in the ferrous condition. With a suitable reducing agent, therefore, a step in the direction of shortening the time had been found. Hydroxylamine hydrochlorate proved to be an ideal reducing agent, and comparative tests showed that the results obtained were the same as with the Lunge method; hence no sacrifice of accuracy.

As regards the amount of ore needed for a sulphur determination, this will largely depend upon the sensitiveness of the balance used, and it may therefore be necessary for some to use as much as 1 grm. of ore, as has also been proposed. With a sensitive balance there is no need of using over 0.5 grm., as Dr. Lunge recommends, and even 0.2 grm. is sufficient for all practical purposes.

I work as follows:—

Two-tenths of a grm. of ore ground until almost free from grit in an agate mortar is treated with a mixture of 5 c.c. concentrated HCl and 15 c.c. concentrated HNO_3 in a small covered beaker, as recommended by Dr. Lunge.

This acid mixture is far preferable to bromine or fuming nitric acid, and has never yet failed to oxidise the whole of the sulphur for this amount of ore. With 0.5 or 1 grm. of ore it not unfrequently fails in this respect, as do also the other two reagents mentioned.

The beaker is placed on the water-bath, and when all action has ceased the cover is rinsed off with distilled water, removed, and the contents of the beaker evaporated to dryness. When dry, moisten with a little water, add 5 c.c. concentrated HCl , and again evaporate to dryness. When dry the second time add 100 c.c. distilled water, 1 c.c. concentrated HCl , and 3 c.c. hydroxylamine hydrochlorate solution containing 1 oz. of the salt in 500 c.c. distilled water. When the iron is reduced—that is, when the solution is colourless—filter and wash with hot water exactly as recommended by Dr. Lunge. Heat the filtrate to *near boiling*, and add 10 c.c. of a 10 per cent BaCl_2 solution. Place beaker on water-bath to settle, then remove to the bench to cool to room temperature before filtering off the BaSO_4 , which, after being washed free from chlorides, is dried, incinerated, and weighed as usual.

It is immaterial, as far as the amount of sulphur precipitated is concerned, whether one uses hot BaCl_2 as recommended by Dr. Lunge, or drops the cold BaCl_2 solution, but the manner of adding the BaCl_2 solution may greatly affect the filterability of the BaSO_4 formed. With the Lunge method one has very little trouble in producing a coarse, easily settling precipitate, due, presumably to the action of the large amount of ammonium salts present, but this precipitate is rather difficult to wash free from chlorides.

With the hydroxylamine method the BaSO_4 is in a much finer state of division, but with proper care it is dense enough for any ordinary filter-paper not too thin. I succeed best when dropping in the cold BaCl_2 solution slowly and with stirring to the almost, but not quite, boiling solution, the whole operation occupying about two minutes. One may transfer the BaSO_4 precipitate to the filter at once without previous decantation with hot water, as it washes very much more readily than does that obtained by the Lunge method. One may carry on any number of simultaneous determinations without fear that any inattention to a funnel at a particular time will cause complications, as may be the case with the Lunge method if one has too many ferric hydrate separations at one time, or if, being called away to attend to some other work for a

little while, the precipitate of ferric hydrate becomes too dry and assumes a lumpy instead of a flocculent appearance on further treatment with water, and which leads to a considerable retention of sulphur, even though by proper care in precipitation with ammonia one has guarded against the formation of baric salts.

The following results were obtained by myself with the hydroxylamine method, and by a New York chemist by the Lunge method.

	Hydroxylamine method.	Lunge's method.
Canadian ore (1) ..	42'40	42'29
" " (2) ..	44'20	44'14
Spanish " ..	46'16	44'19

It will therefore be seen that the two methods give the same result, while that with hydroxylamine is much shorter and more convenient.

THE CHEMICAL ELEMENTS.

A NEW CLASSIFICATION.

By GEO. WOODIWISS.

THE accompanying diagram is designed with the object of illustrating the inter-relationship of the chemical elements as shown by their relative weights. The weights of the elements may be considered from two extremes of temperature; between these two extremes the ratio of weights may be altered by alteration in temperature, but the limit of alteration is arrived at at these extremes. The weight of the elements may be represented in the first extreme by the specific gravity in the atomic state, and in the second extreme in the solid state by the specific gravity at the absolute zero of temperature. And for the purpose of the diagram the latter may be represented by the specific gravity of the elements as compared at 0° C., the figures in brackets indicating comparison in the liquid state.

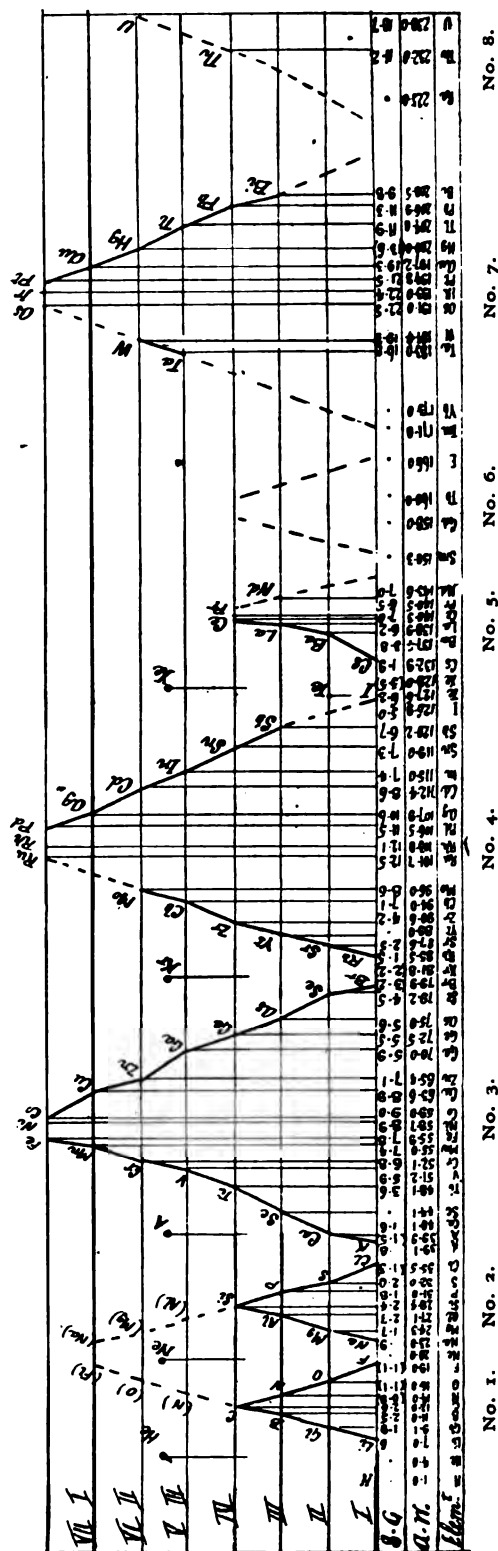
If we form a list of the elements in the order of their atomic weight, by an examination of this alone we find little indication of a periodicity, but if we combine with this a knowledge of the properties of the elements, we are then in a position to prove a periodicity. This has been conclusively shown by Mendeleeff.

If we form a list of the elements in the order of their atomic weight and divide this list into periods as indicated by the specific gravity in the solid state, the analogous elements are shown in an order differing from that of Mendeleeff, but in a clear and remarkable manner.

In the diagram, therefore, the elements are shown horizontally to scale according to their atomic weight, and vertically on eight equidistant lines of valency according to their specific gravity. The diagram is thus divided into a series of eight colonies or tents of the elements; these tents have a most remarkable relationship to each other, and particularly so when taken in evident pairs. Many of the characteristics of the elements are repeated in a more or less degree by those occupying analogous positions. Those noted for their power of occluding gases, for instance, are found at the crown of the tents; copper and silver, the free conductors of heat and electricity, occupy analogous positions, as do the alkali metals, the alkaline earths, and many others. Those elements occupying corresponding positions on opposite sides of the tents have more or less opposite properties, as potassium and bromine, manganese and copper, &c. The elements on the left slope of the tents have increased specific gravity with increase of atomic weight; those on the opposite side have a decrease of specific gravity with the increase of atomic weight. The heavy metals are separated from the light metals, and the non-metals are separated from them both, and each in their respective tent gradually merge the one into the other.

That the specific gravity is a guide as to the formation of these tents may be illustrated by tents No. 1 and No. 2, for by ignoring specific gravity these two may be combined

A DIAGRAM OF ATOMIC WEIGHT AND SPECIFIC GRAVITY OF THE CHEMICAL ELEMENTS.



to form one, as shown by the dotted lines in the diagram, the altered position of the elements being shown in brackets. The tent so formed is in many respects similar to the larger tents; nevertheless, it is still evident that it is quite distinct, is insufficient in width, and could never form one similar to the larger kind. It serves to show, however, that there is a connection between the upper and lower parts of the larger tents; for if we now invert the right-hand side of all the tents (from the base to the seventh line) the elements are then on oblique lines, almost but not quite parallel, and the elements are then in the order of the periodic system of Mendeleeff; thereby it may be shown that in the larger tents the elements in the upper part on the one side are in many ways connected with those in the lower part of the opposite side when taken in the inverse order.

The position of hydrogen is not indicated in the tents, neither are the gases helium, neon, argon, krypton, and xenon; these inert gases are shown according to their atomic weight, and, with the exception of argon, find a position between the tents, and as they are not known to have chemical affinity they are not shown on any line of valency.

THIRTEENTH ANNUAL REPORT
OF THE COMMITTEE ON ATOMIC WEIGHTS.
DETERMINATIONS PUBLISHED IN 1905.*

By F. W. CLARKE.

(Concluded from p. 203).

Metals of the Rare Earths.

A CONSIDERABLE number of memoirs upon the rare earths have appeared in the course of the year, with incidental data on the subject of their atomic weights. In only two of them was the determination of atomic weight the principal feature, the paper by Urbain (*Comptes Rendus*, xl., 583) on gadolinium being the most important. Urbain prepared his material by many fractionations of gadolinium-nickel nitrate, and analysed a number of distinct preparations of the octohydrated gadolinium sulphate. According to Eberhard (*Zeit. Anorg. Chem.*, xlv., 375), who has studied several rare earths spectroscopically, Urbain's material for samarium, gadolinium, and europium was exceedingly good, if not absolutely free from admixtures. Bettendorff's samarium preparation contained other earths.

I	Li	Na	K	Rb	Cs	—	—	—	Cu	Ag	Au	—	I
	Fl	Cl	Br	I	—	—	—	—	Mn	—	—	—	VII
II	Gf	Mg	Ca	Sr	Ba	—	—	—	Zn	Cd	Hg	—	II
	O	S	Se	Te	—	—	—	—	Cr	Mo	W	U	VI
III	B	Al	Sc	Yt	La	—	—	—	Ga	In	Tl	—	III
	N	P	As	Sb	Nd	—	Bi	—	V	Nb	Ta	—	V
IV			Ti	Zr	Ce	—	—	Th	Fe	Ru	Os	—	
	C	Si			Pr	—			Ni	Rh	Ir	—	IV
			Ge	Sn	—	—	Pb	—	Co	Pd	Pt	—	

An item of interest is the systematic arrangement of the non-metallic elements. The first four tents contain five, four, three, and two of these elements respectively, in each case the more active element being found at the base and on the right-hand side of the tent; this symmetrical arrangement and gradual decrease suggesting a further decrease in the remaining tents.

By taking consecutively from tent to tent, the elements from analogous positions, the accompanying table is formed.

It is of interest to note the lowering of the melting-point from lithium to caesium, and the rise in the melting-point from fluorine to iodine, the one being in the reverse order to the other.

It will be noticed there is no anticipation of a noble metal intermediate between silver and gold, nor a set of three intermediate between palladium and platinum, but there is a greater anticipation of the rare earths, &c.

Royal Institution.—On Saturday, May 19th, at 3 o'clock, Sir James Dewar will deliver the first of a course of two lectures at the Royal Institution, on "The Old and the New Chemistry." The Friday Evening Discourse on May 18, will be delivered by Professor Arthur Schuster on "International Science."

For gadolinium, Urbain's data are as follows, calculated with $H = 1.007$ and $S = 32.006$:—

Fraction.	Weight $Gd_2(SO_4)_3 \cdot 8H_2O$.	Weight Gd_2O_3 .	Atomic weight.
18.	1.9256	0.9350	157.35
19.	1.9749	0.9589	157.35
19.	1.9975	0.9698	157.32
20.	2.1083	1.0231	157.15
20.	1.8993	0.9214	157.04
21.	2.2065	1.0707	157.13
21.	1.9535	0.9479	157.12
22.	2.2008	1.0685	157.32
22.	2.2482	1.0914	157.28
23.	2.1932	1.0646	157.25
35.	2.0551	0.9974	157.19
36.	2.1555	1.0469	157.45
37.	2.2277	1.0807	157.04
38.	2.2559	1.0946	157.11
39.	2.2523	1.0939	157.45

Mean 157.24

The value adopted by Urbain from Fractions 18 to 23 is $Gd = 157.23$.

* From the *Journal of the American Chemical Society*, xxviii., No. 3.

The paper by Brill (*Zeit. Anorg. Chem.*, xlvii., 464) describes an attempt to determine atomic weights on very small quantities of material by means of the micro-balance invented by Nernst. In each case a few m.grms. of a sulphate, previously heated to 480°, was ignited to oxide. At 480° acid sulphates were destroyed and the normal salts were stable. The results obtained may be summarised as follows:—

Yt.	Er.	Yb.	La.	Sm.	Nd.
89.7	165.6	173.2	139.5	151.2	141.8
89.5	167.5	172.2	139.8	150.5	143.0

These figures, of course, are only approximations; but they indicate the convenience of the method for the ordinary rough determinations which are needed in the identification of the earths.

Feit and Przibylla (*Zeit. Anorg. Chem.*, xliii., 202), in an investigation of the monazite earths, made several determinations of atomic weights by a volumetric method. The weighed oxides were dissolved in a known quantity of standard sulphuric acid, and the excess of the latter was ascertained by titration, with methyl- or ethyl-orange as indicator. For lanthanum, in a single experiment, the value 139.0 was found.

For neodymium and samarium the following results were obtained:—

	Nd.		Sm.
	144.51		151.20
	144.48		151.25
	144.53		151.13
Mean ..	144.5	Mean ..	151.2

Matignon (*Comptes Rendus*, cxli., 1230) has found that samarium sulphate heated to about 1000° is converted into a stable basic salt, Sm_2SO_6 . He proposes to use this transformation as a means of determining the atomic weight of samarium. In one experiment, 0.7325 gm. of $\text{Sm}_2(\text{SO}_4)_3$ calcined at a dull red heat gave 0.5335 of the basic compound. Hence $\text{Sm} = 150.6$.

According to Urbain (*Bull. Soc. Chim.*, [3], xxxiii., 403), in a communication to the Chemical Society of Paris, the following atomic weights are approximately true:—Terbium, 159.5; dysprosium, 162–163; holmium, 140. In a later paper he assigns the value 159.2 to terbium (*Comptes Rendus*, cxli., 521). For an impure terbium preparation Feit found the value 158.6 (*Zeit. Anorg. Chem.*, xliii., 280). Emma Potratz (*CHEMICAL NEWS*, xcii., 3), from a preparation of terbium sulphate, found $\text{Tb} = 153.9$, and from another sample 154.2. From the formate she obtained the figure 154, and from the acetate 153.1. No details are given, but another report is promised in the future.

Miscellaneous Notes.

From the density and critical constants of sulphur dioxide, Guye has computed its true molecular weight (*Comptes Rendus*, cxl., 1241); $\text{SO}_2 = 64.065$, and therefore $\text{S} = 32.065$. The molecular weight of argon, determined in the same way, is 39.866. Jaquerod and Scheurer (*Comptes Rendus*, cxl., 1384), from the compressibility and density of SO_2 , determine its molecular weight as 64.036, whence $\text{S} = 32.036$. Guthe (*Bull. U.S. Bureau of Standards*, i., 349), in a paper upon the silver coulometer, has discussed the electro-chemical equivalent of silver. There is also a memoir by Reuterdahl on electro-chemical equivalents and atomic weights (*Trans. Amer. Electrochemical Soc.*, vii., 187).

Makower has attempted to determine, from their rates of diffusion, the molecular weights of the radium and thorium emanations (*Phil. Mag.*, [9], ix., 56). For the radium emanation he finds the values 85.5, 97, and 99, assuming the substance to be monatomic. The thorium emanation is but slightly different. Makower suggests that the emanation may fill the vacant place in the periodic table between molybdenum and ruthenium. The atomic

weight of radium itself has been discussed by Jones (*Am. Chem. Journ.*, xxxiv., 467), who, from a critical examination of all the evidence, is inclined to favour the higher of the two rival values, namely, $\text{Ra} = 258$.

On the calculation of atomic weights, there is an interesting paper by J. Meyer (*Zeit. Anorg. Chem.*, xliii., 242). An important suggestion by Luther (*Zeit. Elektrochem.*, xi., 273) is to refer combining weights, through the aid of Faraday's law, to the C.G.S. system of units. In this way the question of standards might be settled and a rational table devised.

A SYSTEM OF QUALITATIVE ANALYSIS INCLUDING NEARLY ALL THE METALLIC ELEMENTS.*

PART II.—ANALYSIS OF THE TUNGSTEN GROUP.

By ARTHUR A. NOYES.

(Continued from p. 207).

Test Analyses.

At different stages in the development of the process above described, numerous analyses were made to test its efficiency. Nearly all of those which were made after integral parts of the procedure assumed final form are reproduced in the tables below. In these tables the numbers in each vertical column show the weights in m.grms. of the various elements which the solution submitted to analysis contained. The results of the tests for each element are shown by the letters following these numbers. That the result was satisfactory is indicated by the letter S; that is, when the element was present, that the test for it, however small, was unmistakable and therefore conclusive; and when the element was absent, that a good blank test was obtained. When the test was very small in comparison to the quantity of the element present, though still unmistakable, or when in its absence (especially in the niobium and tantalum tests) there was obtained not a perfect blank but yet one good enough to warrant the conclusion that less than one m.grm. is present, this is sometimes indicated by the symbol S—. When in the presence of the element the test failed, or in its absence a result was obtained that might be thought to indicate its presence, the letter F is used. When the result was doubtful or inconclusive, owing, for example, to the test being obscured by some other element, this is indicated by the letter D. When the test was not tried, if the element was present, a dash is used in place of a letter, or, if the element was not present, the whole space is left blank.

The first series of twenty-three analyses were made by Mr. D. P. Smith, at a time when the procedure through the analysis of the titanium, tantalum, and niobium hydroxide precipitate was in nearly its final form, but when the tin and tungsten were still being separated by heating the moist, precipitated sulphides with strong hydrochloric acid. Therefore, owing to the solubility of tungsten sulphide under these conditions (see G. D., § 10), the failures to detect tungsten in the presence of much tin in these analyses do not show a defect in the final procedure. In all of the analyses of this series, before testing for tungsten, the sodium carbonate solution (P. 40) obtained from the filtrate from the sulphuric acid precipitate was united with an ammonia solution of the residue left upon treating the sulphides with nitric acid (P. 43), and the mixed solutions were then evaporated with nitric acid to separate the yellow tungstic acid. The results given for the niobium tests are always those obtained in the second test with mercuric chloride after the fusion with sodium carbonate and sulphur, when a precipitate obtained in the first test made this fusion necessary.

A second series of analyses (Nos. 24–29) were made by Mr. F. M. Eaton, who had no previous experience in con-

* From the *Technology Quarterly*, xvii., No. 3.

FIRST SERIES.

T. A. No.	1 (a)	2	3	4	5	6	7	8
Analysis begun at P. ...	3	3	3	33	33	33	33	33
Ta	0 S	0 S	2 F	2 S	2 S			2 S
Ti	500 S	100 S	300 S	300 S	300 S	300 S	300 S	300 S
Nb	0 S	0 S	1 F			0 S—	0 S	0 S—
Sn	1 F	1 S	300 S	3 S	300 S			2 S
W	1 F	2 S	1 F	300 S	5 F	300 S	5 S	300 S

T. A. No.	9	10	11	12	13	14	15	16
Analysis begun at P. ...	33	33	33	33	33	33	33	5
Ta	0 D (b)	2 S	0 S—(b)		2 S	100 S	2 S	2 S
Ti	300 S	3 S	300 S	200 S	300 S	2 S		2 S
Nb	2 F	0 S	5 S	2 S (c)		2 S (c)	100 S	0 F (d)
Sn	300 S	300 S	300 S					
W	2 F	300 S	5 F					3 F (e)
Mo								500 S

T. A. No.	17	18	19	20	21	22	23
Analysis begun at P. ...	31	33	33	33	33	33	33
Ta							
Ti	300 S	5 S		5 S	300 S	300 S	300 S
Nb	0 S		5 S				
Sn		300 S	300 S				
W				300 S	5 F	5 S	10 S
Mo	300 S						

SECOND SERIES.

T. A. No.	24	25	26	27	28	29
Analysis begun at P. ...	31	31	31	31	31	33
Sb	3 S	2 S	1 S	3 S		
Ta	3 S	2 S	1 S	3 S	0 S	2 S
Ti	3 S	1 S	1 S	3 S	2 S	300 S
Nb	3 S	2 F	1 S—(h)	3 S	2 F (i)	0 S (g)
Sn	3 F	2 F	1 F	3 S	2 F	2 F
W	3 F	300 S	1 F	3 S (f)	2 F	2 F
Mo					300 S	

THIRD SERIES.

T. A. No.	30*	31*	32*	33*	34	35	36	37*	38	39
Analyst	D. P. S.	F. M. E.	F. M. E.	F. M. E.	D. P. S.	D. P. S.	D. P. S.	F. M. E.	D. P. S.	D. P. S.
Analysis begun at P. ...	31	31	33	31	31	31	31	31	31	35
Sb	0 S	400 S		0 S			400 S			
As	2 S			2 —					100 S	
Ta	0 F	0 D (k)	2 S—(l)	2 S	0 S	0 D (b)	2 S	2 S	0 S	
Ti	2 S	2 S	400 S	400 S	2 S	400 S	2 S	2 S	2 S	400 S
Nb	2	2	0	0	0	0	2	2	0	0
Sn	400 S	2 F	2 F	4 F	2 F	4 F	4 S	4 F	400 S	
W	2	2	4	400	4	4	4	4	4	
Mo		0 S	2 S	2 S	400 S	400 S	2 S	400 S		200 S

FOURTH SERIES.

T. A. No.	40	41	42	43	44	45	46	47
Analyst	D. P. S.	D. P. S.	D. P. S.	F. M. E.	F. M. E.	F. M. E.	F. M. E.	D. P. S.
Analysis begun at P. ...	33	33	33	35	31	35	35	35
Sn	300 S	300 S	1 S	30 S	8 S			
W	3	1	300	8	8	4	2	2
Mo					400 S	100 S	100 S	100 S
PO ₄				20 S				

nection with this process, and who was given no information in regard to it other than that contained in a copy of the "Procedure and Notes." The procedure was then in final form, except that the hydrochloric acid solution of the ignited sulphides was tested for tin by adding it directly to mercuric chloride, instead of first reducing it with zinc, which accounts for the repeated failures to detect small quantities of tin (see P. 42, N. 1). The results with niobium were those obtained in the second mercuric chloride test after the fusion with sodium carbonate and sulphur, except where otherwise indicated by foot-notes. The sulphuric acid precipitate and the filtrate from it were separately tested for tungsten.

The third series of analyses (Nos. 30—39) were made according to the final procedure in every respect, in part by Mr. D. P. Smith and in part by Mr. F. M. Eaton, as indicated by the initials at the head of the columns. As a matter of information, the results of both the first and second mercuric chloride tests for niobium are recorded, but it is only the second one that is really significant of the presence or absence of that element, except when the first test gives a negative result. The results of testing for tungsten both the sulphuric acid precipitate (by P. 41 and 43) and the filtrate from it (by P. 40) are also shown, but the detection of it in either place is, of course, a satisfactory result. In those analyses to the numbers of which a star is attached the contents of the mixture were unknown to the analyst in advance. Such unknown mixtures were not analysed in greater number, because to a person having any experience with the process the interpretation of the tests is scarcely ever doubtful; and as long as the procedure is in process of development, it is advantageous that the analyst know immediately of failures to secure the right result.

The fourth series of analyses (Nos. 40—46) were also made after the procedure was fully developed, but they have reference only to the detection of tin, tungsten, molybdenum, and phosphate in the presence of one another.

In order to summarise the results of these analyses, and form thereby a judgment in regard to the efficiency of the procedure, each element will be considered separately.

Notes to Table.

- (a). This mixture also contained 1 m.grm. Sb and 1 m.grm. Mo, and these were detected in the H_2S precipitate.
- (b). A small precipitate (very slight in T. A. 11) was obtained, owing to a little tantalum in the titanium used. To show the presence of this impurity, 400 m.grms. Ti were alone put through P. 33, 35, and 36; a precipitate corresponding to that given by 2 m.grms. Ta was obtained.
- (c). This was the result even after the fusion with Na_2CO_3 and S, which was made to see whether it would result in loss of the niobium.
- (d). In this analysis the $(NH_4)_2S$ precipitate was not redigested with NH_4OH , as directed in P. 35, when much molybdenum is present.
- (e). H_2SO_4 was not used, as directed in P. 43, to cause the tungsten to separate.
- (f). Tungsten was detected only in the filtrate.
- (g). Fusion with Na_2CO_3 and S was omitted, since no precipitate formed in first $HgCl_2$ test.
- (h). Fusion with Na_2CO_3 and S was omitted, since the precipitate in first $HgCl_2$ test was too slight.
- (i). There was a very slight turbidity in the first $HgCl_2$ test.
- (k). A light, flocculent precipitate separated on standing.
- (l). A light, flocculent precipitate first separated; but upon repeating the treatment of P. 36, it assumed the characteristic appearance of the tantalum precipitate.
- (m). The reduction test with Zn at end of P. 40 was not tried, which may have been necessary, since the

arsenic, if present in this solution, would have prevented the separation of tungstic acid.

- (n). No yellow H_2WO_4 separated, but a decided blue coloration was obtained with Zn.

(To be continued).

A REVISION OF THE ATOMIC WEIGHTS OF SODIUM AND CHLORINE.*

By THEODORE WILLIAM RICHARDS
and
ROGER CLARK WELLS.

(Continued from p. 203).

THE OCCLUSION OF DISSOLVED SUBSTANCES BY ARGENTIC CHLORIDE.

It is well known that all precipitates have a tendency to carry down with them other substances from the solution in which they are formed (Jannasch and Richards, *Journ. Prakt. Chem.*, 1889, xxxix., 321; Schneider, *Zeit. Phys. Chem.*, 1892, x., 425; Richards, *Proc. Amer. Acad.*, 1900, xxxv., 377; *Zeit. Phys. Chem.*, 1903, xlv., 189). According to the nature of the precipitate, the mechanism of this contamination varies. A basic precipitate such as ferric hydroxide is likely to carry down traces of acid in the form of basic salt; an acid precipitate such as silica is likely to carry down traces of base; many substances are contaminated in the act of precipitation by the admixture of isomorphous substances in solid solution; all or nearly all crystals contain within themselves minute cells inclosing mother-liquor, and very finely divided precipitates, because of their large surface, pertinaciously adsorb in varying amount impurities from the supernatant solution. Obviously contamination from any of these sources must be scrupulously avoided in accurate work, and the peculiar qualities of each precipitate under investigation must be studied.

As has been said, argentic chloride was known by Stas to occlude argentic nitrate, which is easily detected by its blackening effect upon fusing the contaminated chloride. (Richards, *Proc. Amer. Acad.*, 1893, xxix., 76. The blackening occurs in full measure only when the argentic nitrate is occluded from aqueous solution. Perfectly dry argentic nitrate fused with dry chloride is but slightly decomposed, and the fused mass is merely grey, not black. The probable explanation is obvious). This test is a delicate one. Stas found that upon working with solutions of argentic nitrate weaker than decinormal, the occlusion was so slight as to be negligible; therefore in his early experiments he took great pains never to use solutions stronger than this. Later, he seems to have overlooked this precaution, since he used much more concentrated solutions. Although realising that argentic nitrate is occluded by the precipitate, Stas seemed never to have guessed that other salts might also be taken up in this way. Evidently the question was one which needed further investigation, and the outcome of the investigation is recorded below.

It was convenient at first to determine whether or not the occlusion of sodic nitrate or sodic chloride would also affect the appearance of the silver salt on fusion, in order to discover an easy qualitative test for the presence of either. As a matter of fact, the former salt was found by actual admixture to colour the fused silver chloride a pale buff, rendering it opaque; while the latter had no perceptible effect upon its appearance. Thus Stas could not have detected the presence of sodic chloride in his precipitate by the appearance; whether fused or unfused. An impurity of argentic nitrate being thus the most easily detected among the possible impurities, the next step was

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to use the contamination with this salt as a means of testing the properties of the precipitate.

It was early found that upon shaking pure argentic chloride with a solution of argentic nitrate, the latter is adsorbed upon the surface of the precipitate. If a solution containing argentic nitrate and the curdy precipitate is divided into two equal volumes, one containing the precipitate and one clear, and if the precipitate is then carefully washed, more argentic nitrate will be found in the portion which contained the precipitate than in the other portion. A further evidence of this adsorption is found in the fact that the theoretical amount of washing is especially inadequate to remove the last traces of adhering electrolyte.

These experiments show not only that the nitrate is adsorbed, but also that some at least of the adsorbed salt may finally be washed off.

In view of this result, it seemed probable that the reason why silver chloride occludes silver nitrate in the act of precipitation is because the former adsorbs the latter during this process, and the adsorbed material is then covered up by newly-formed precipitate. The more dilute the precipitating solutions, the less considerable would be the adsorption.

The peculiar curdy, flocculent form of this precipitate suggested that since the material is so flexible, long-standing and gentle agitation with pure water might allow the occluded material to escape, although from a more rigid precipitate this escape is known to be impossible. With this possibility in view, a number of experiments were made in the following manner:—Argentic chloride was precipitated from several portions of a concentrated solution of the nitrate, thus occluding much (in some cases as much as 0.1 per cent) of this salt. The effort was then made to wash these precipitates; and the possibility of thorough washing was found to vary greatly with the treatment of the precipitate. In those cases where the precipitate had remained for two or three days in contact with a mother-liquor rich in silver, it was found much more difficult to remove the nitrate than in cases where the washing was more prompt. It seems as if the innermost cells of the precipitate are closed upon standing during the well-known contraction which then occurs. In one case where normal solutions had been used in precipitation, six prompt, gently-agitated washings with pure water were enough to remove practically all the occluded material, and yield a chloride which fused without blemish; but this praiseworthy behaviour was the exception rather than the rule. In this case the innermost cells must have been cleaned before they closed during the compacter aggregation of the precipitate; but usually traces of silver nitrate were enclosed, and when they were once enclosed were beyond the reach of even a dozen washings. Therefore more dilute solutions of argentic nitrate were used in precipitating, in order to find the limit beyond which a satisfactory precipitate was certain. Third normal solutions were found to be still slightly too concentrated, but fifth normal solutions always yielded a satisfactory result, if speedily but gently washed six times, and then occasionally agitated in pure water in order to dissolve out the last trace of nitrate. In previous determinations in this laboratory still less concentrated solutions (tenth normal) were used, so that no correction need be applied to those determinations on this account. (See, for example, Richards, *Proc. Amer. Acad.* 1893, xxix., 76). This further dilution is necessary unless the curdy precipitate is speedily washed or agitated with a solution containing no excess of silver.

Having thus obtained light on the mechanism of the occlusion of argentic nitrate, we turned our attention to the possibility of occluding sodic chloride. Here, as had been said, no qualitative experiment can conveniently prove the presence of the impurity. Hence recourse was had to a quantitative experiment. An amount of pure salt known by previous experiment with very dilute solutions to be exactly equivalent to a given weight of pure silver

was carefully weighed out, and the solid was dropped into a fairly concentrated solution of the nitrate, according to Stas's later method of procedure. After two hours' thorough shaking and a day's standing, which Stas had always given, it was found that 0.014 per cent more of salt had to be added in order to attain exact equivalence, showing that this amount of salt remained occluded by the precipitate even after the shaking and standing. Agitation for another day leached out about one-half of the occluded salt, but seven days' soaking and agitation were required before the full amount (0.014 per cent) of extra silver nitrate was needed to attain equivalence with the salt dissolved out of the precipitate. Subsequently, in several days no more salt appeared; hence the final condition must have been reached. Since probably some silver nitrate also was occluded, it is clear that these figures represent a minimum value, and the certain error introduced by Stas's method of procedure was demonstrated. The reason why he obtained consistent results was simply because he used exactly the same method in each analysis.

Obviously, then, a solid salt is not to be dropped into a precipitating solution—indeed, such a proceeding is so repugnant to the instinct of a precise quantitative analyst that it is surprising Stas should have been guilty of it. But how dilute must the precipitating sodic chloride be in the present case in order to avoid the danger? Careful experiments showed that with fifth normal solutions, as in the case of silver, such slight occlusion as existed was quickly removed by gentle agitation, and after a short time the mixture showed a constant equilibrium point. Hence in our final experiments the solution was always as dilute as this. In previous similar work in this laboratory, the solutions of halide were rarely stronger than fifth normal, and never much stronger, so that the error from this source must have been very small.

It might be supposed that when the sodic chloride and argentic nitrate are too dilute to be carried down with the precipitate, sodic nitrate is not likely to be carried down in important amounts. For if both silver and chloride are fifth normal, the resulting sodic nitrate must be tenth normal, a dilution at which even silver nitrate is not sensibly occluded. Nevertheless, as a matter of fact, the final experiments yielded quantitative evidence of the occlusion of a trace of this salt, amounting to 0.005 per cent of the weight of the precipitate. This is probably because the precipitate was obliged to stand for some time in the solution, and it is doubtful if this source of error could be wholly avoided.

From these experiments it appears that the degree of dilution at which occlusion ceases to be seriously troublesome is about the point at which most physical chemists agree that the realm of dilute solutions begins. Hence it would seem that in these cases of adsorptive occlusion the undissociated salt is that which is occluded, in confirmation of other work (Richards, MacCaffery, and Bisbee, *Proc. Amer. Acad.*, 1901, xxxvi., 377).

Obviously the occlusion of argentic nitrate, or of sodic chloride, would affect the results of the analysis, no matter whether silver or argentic chloride served as the weighed standard wherewith to compare the weighed sodic chloride, although more seriously in the former case than in the latter. On the other hand, the occlusion of sodic nitrate could only affect the result when the argentic chloride was weighed; its occlusion would have no effect on the amount of silver required to precipitate the chlorine. The possible effect of these disturbing influences will be alluded to again during the recital of the quantitative operations.

It is needless to say that these experiments were carefully carried on in complete darkness, in order to avoid the known disturbing effect of photochemical action. The temperature was usually 20° C. Undoubtedly this condition is important, since the aggregation of argentic chloride is greatly affected by change of temperature.

(To be continued).

PROCEEDINGS OF SOCIETIES.

PHYSICAL SOCIETY.

Ordinary Meeting, April 27th, 1906.

Dr. C. CHREE, F.R.S., Vice-President, in the Chair.

MR. W. B. CROFT read a paper on "*Some Simple Questions on the Images of Microscopes and Telescopes.*"

It may have been noticed that when a microscope is focussed visually, an image is formed on the focussing-glass of a camera, into which the microscopic eye-piece is inserted after removing the camera-lens. This image remains more or less in focus at variable positions of the camera-screen. Although it is not always perhaps true, yet it is surprising how often the pencil emerging from a microscope eye-piece behaves like a single concentrated line of light. A mounting of the proboscis of a fly was focussed visually, then a camera was put on the end of the microscope, and the screen was racked out to six positions over a range of about six inches. At these positions six photographs were taken, of which the two smallest pictures were inferior to the others. It was claimed that this defect was due to imperfect exposure and uneven illumination. If the visual focussing had made pencils slightly diverging instead of parallel, the larger pictures should have been the more imperfect. These pictures were obtained with a 1-inch objective. Next, a diatom, *Triceratium favus*, was photographed with a $\frac{1}{2}$ -inch objective. With a visual focussing two photographs at different distances were taken; these were similar to one another and to what was seen. The same diatom then had a slightly different visual focussing, with the same result as before in the two pictures. Finally, two diatoms with fine markings were focussed visually and at two positions; the markings came out clearly in the photograph. The object of showing the results was not to make out that the emergent pencil was ever actually, or always approximately, a single line of light, but to intimate how often the author had found, when projecting from an optical eye-piece, that no change can be detected in the definition of the image as the screen of the camera is moved. If a camera-lucida is placed on the eye-piece, the image of a stage-micrometer can be thrown on a scale at 10 inches distance or at 40 inches distance. The parallel rays emerging from the eye-piece give the image of a point along a direction, at no definite position. The image can be imagined at 40 inches distance as easily as 10 inches.

Mr. CROFT also showed some photographs taken from sections of the human eye; he indicated that a divergent pencil from a small aperture or from a convex reflecting surface of large curvature will give the Purkinje figures as bright radiating lines, whereas the usual method of sending light through the side of the sclerotic gives them as shadows.

Several different specimens were shown of magnetic oxide of iron and magnetic sulphides of iron. The power of nickel and cobalt to receive permanent magnetism was illustrated with a compass-needle of nickel.

Mr. A. P. TROTTER, referring to the fact that when taking microscopic photographs the screen could be placed at any distance from the eye-piece of the microscope, pointed out that this was not the case when using an astronomical telescope, the screen having to be placed in a definite position to obtain a sharp focus.

Prof. H. L. CALLENDAR, referring to the fact that when the microscope used in the author's experiments was focussed visually, it also formed sharp images on a screen at any distance, suggested that this might be due to some peculiarity in the eye of the observer.

Mr. C. V. BOYS gave a demonstration of the construction and action of his gas calorimeter described in the *Proceedings of the Royal Society* for January, 1906.

This is the instrument which is being used for testing the calorific value of London gas under the provisions of the London Gas Act, 1906.

Mr. J. MORROW read a paper "*On the Lateral Vibration of Bars Subjected to Forces in the direction of their Axes.*"

In the paper a statement of the general problem of the lateral vibration of rods is first made. The rods are assumed to be subjected to an axial tensile or compressive force, and the differential equation given may be applied to bars of uniform or varying sectional area, and to cases in which the bar carries one or more loads concentrated at different points in its length. In considering any special problem we may either start with this general equation, or we may write out a particular differential equation suitable to the conditions of the case in question only. Three cases of unloaded bars are dealt with, namely, those under the following end-conditions—"supported-supported," "clamped-clamped," and "clamped-supported." Expressions are obtained from which the frequencies may be calculated, and the results are stated in a form such that the determination of stresses, terminal couples, &c., may be easily made. The case of greatest interest is that of a stretched bar clamped at each end. Approximate solutions of this problem have been arrived at by both Seebeck and Donkin. These are on the assumption that the vibration is but slightly affected by the rigidity of the material. An assumption of a very different character, and one generally fulfilled in structural work, is made in this paper, namely, that the longitudinal force is not very great. Solutions are thus found for the period of the fundamental or any harmonic. Two other methods are applied to the solution of the same problem. These are the method of successive approximation, and the approximate "energy-method" of Lord Rayleigh. Both these methods are free from the assumptions mentioned above, and give results closely agreeing with that first obtained. These results may be applied in problems of long struts or tie-rods as distinct from the long and fine wires of acoustics. The cases next dealt with are those of bars of negligible mass, carrying a concentrated load, and subject to longitudinal force. Expressions are obtained for the curve of the bar at any instant, the couples at the end and the periods of vibration. It is found throughout that, when the axial force is compressive, there is a certain limiting load at which vibration ceases. The amount of this load varies with the conditions at the ends of the rod, and the expression for it agrees with that given by Euler for the elastic stability of a long column. The paper closes with an application of the solutions to some static problems of loaded beams. By a simple change of notation the expressions for the displacements of such bars replace those for the types in the vibration problems. Several results not hitherto recorded may be so obtained.

FARADAY SOCIETY.

Ordinary Meeting, April 10th, 1906.

Prof. A. K. HUNTINGTON in the Chair.

MR. F. W. HARBORD communicated to the meeting papers by Messrs. Keller, Stassano, and Gin. The paper by CH. A. KELLER was entitled "*Electrothermics of Iron and Steel.*"

The author deals with the present position of his processes; he describes the electrical steel plant which Messrs. J. Holtzer and Co. have just installed in their works at Unieux (Loire). This is a 1500 H.P. plant, and will utilise in a single furnace the current from a 20,000 ampere Westinghouse alternator. The furnace, which rests on a steel cradle and can be tilted, weighs about 50,000 kilos; the various mechanical and electrica controls are obtained by hydraulic motors. The steel

obtained from a Siemens-Martin furnace will be run into the electric furnace immediately after the oxidising melt, and for the remaining operations of deoxidising and refining, the current exclusively will be used. It will be possible to operate on 8000 kilos. of metal at one time, and the operation will be repeated three or four times in the twenty-four hours. The steel produced by this furnace is intended for cannon and projectiles. The author promises at a future date to furnish details of the results obtained.

Some experiments made at Livet comparing the metal obtained in a 1000 H.P. Keller furnace with that obtained in the blast-furnace are further described, and curves are given showing the proportions of carbon and silicon in castings produced in both ways. It appears that the Keller furnace allows of ready desulphurisation and of easier variation in the proportions of the above elements than does the blast-furnace; it would thus be advantageous for the production of soft castings and castings for mill work. Tests on some of these electrothermic castings are described.

The author has just set up at Livet a 2000 H.P. furnace utilising a current of 25,000 amperes, and capable of producing 20 tons of castings in twenty-four hours. This will be used specially for the industrial working of the process. Further details respecting the new furnace are promised.

Mr. W. MURRAY MORRISON referred to the freezing of the metal in the central passage in the first furnace that had been erected, and asked for further information regarding the easy variations in silicon content claimed by the author.

Mr. F. W. HARBORD said the freezing trouble was probably only due to the special conditions of the first tests recorded, and in any case could be avoided by coupling two or four furnaces together. Regarding variation of silicon content, as a rule this was easily brought about by adding a little silicon pig, but it might conceivably, under special circumstances, be useful to vary it in the way described for the production of good castings.

Prof. A. K. HUNTINGTON described some experiments he carried out in 1881 with the Siemens electric furnace in which he was able to vary the silicon content from $\frac{1}{4}$ to 9 per cent. He had also succeeded in volatilising copper and in making solid tungsten.

The paper contributed by Cav. Megg. ERNESTO STASSANO was entitled "*Nois on the Rotating Electric Steel Furnace in the Artillery Construction Works, Turin.*"

The furnace described and illustrated in the paper is being installed by the "Forni Termoelettrici Stassano" Company for the Italian War Office. It is of the author's well-known arc type, and absorbs 140 kilowatts yielding 2400 kilos. of steel in twenty-four hours. The current is a rotatory one with 80 volts between each phase. The consumption of electrodes is under 5 kilos. per ton of steel, and the cost of renewing the refractory covering of the furnace 10 francs per ton of metal made. The furnace is principally used for refining pig-iron and smelting scrap. The product ordinarily made is used for artillery projectiles.

Mr. W. MURRAY MORRISON thought the efficiency would not be so great as with the Keller or Héroult type.

Prof. A. K. HUNTINGTON asked whether there would not be greater consumption and wear of electrodes if they were touching. He did not consider the process offered any special advantages for ordinary steel making.

Dr. H. BORNs asked whether Stassano always used very pure iron ore from Elba and other Italian mines.

Mr. CHARLES WEISS pointed out that the paper did not state whether steam or water power was used in the Turin Works. He thought that in countries like South America, where water power was available and there was plenty of rich ore, it might be useful to smelt copper ores in the electrothermic furnace.

Mr. F. W. HARBORD said the efficiency as stated was somewhat greater than in the other furnaces. For special castings there might be advantages in heating by radiation

only. The figures given for carbon consumption were smaller than in the Keller or Héroult furnaces. He thought the rotation was the weak point in the furnace.

With regard to the general question of electric steel smelting, a maker had recently told him that he had sold 300 tons of such steel as crucible steel, and the buyers were so pleased with it that they were asking for more. That was strong evidence that good steel could be made in the electric furnace, but it was only high-priced steel that could be commercially made (in England) by such means.

Mr. Harbord then read M. GUSTAVE GIN's "*Note on Recent Developments in the Gin Electric Steel Furnace.*"

The author's canal type of furnace is now installed at the Plettenberg Works, Westphalia, of which illustrations are given in the paper, but it is not stated which particular type of furnace has there been experimented with. The following types are described:—

1. *Furnace with Canals and Chambers.*—Unlike in the canal form originally suggested, the electrothermic heating and refining now take place separately, the latter in chambers which communicate with each other and with the leads by the canals, now used exclusively for heating. The construction of a 7200 kilowatt furnace, utilising 60,000 amperes at 120 volts and designed to yield over 350 tons in the twenty-four hours, is described in detail. The author claims that the furnace combines the advantages of the Martin and Talbot furnaces, the separation of the operations making exact control of composition possible.

2. *Combination Furnace.*—This consists of a melting and oxidising crucible, a deoxidising and re-carburetting compartment, and a final mixing chamber, and is fully described and illustrated in the paper.

3. *Induction Furnace.*—The author's suggestions for various types of induction furnace are described. The use of multiple canals is proposed, so that if steel is being produced, oxidation can be carried out in one canal and reduction in the other. In another suggested type, more intimate circulation and mixing of the product is brought about by means of lateral conduits connecting the respective ends of the open flues of which the channel crucible is made up.

The paper by Mr. H. S. COLEMAN entitled "*Notes on the Cleaning of Work by means of the Electric Current*" was communicated by Dr. F. MOLLWO PERKIN. (See p. 167).

ROYAL INSTITUTION.

Annual Meeting, May 1st, 1906.

THE DUKE OF NORTHUMBERLAND, K.G., in the Chair.

THE Annual Report of the Committee of Visitors for the year 1905, testifying to the continued prosperity and efficient management of the Institution, was read and adopted, and the Report on the Davy-Faraday Research Laboratory of the Royal Institution, which accompanied it, was also read.

Forty-five new Members were elected in 1905. Sixty-three Lectures and nineteen Evening Discourses were delivered during the year. The Books and Pamphlets presented amounted to about 254 volumes, making with 697 volumes (including Periodicals bound) purchased by the Managers, a total of 951 volumes added to the Library in the year.

Thanks were voted to the President, Treasurer, and the Honorary Secretary, to the Committees of Managers and Visitors, and to the Professors, for their valuable services to the Institution during the past year.

The following gentlemen were unanimously elected as Officers for the ensuing year:—

President—The Duke of Northumberland.

Treasurer—Sir James Crichton-Browne.

Secretary—Sir William Crookes.

Managers—Sir William de W. Abney, The Rt. Hon. Lord Alverstone, The Rt. Hon. Earl Cathcart, Dr. A. H. Church, Dr. F. Elgar, Dr. D. W. C. Hood, Mr. M. Horner, Sir William Huggins, The Rt. Hon. Lord Kelvin, Mr. H. F. Makins, Dr. Ludwig Mond, Sir R. Douglas Powell, Bart., The Rt. Hon. Lord Sanderson, Mr. Alexander Siemens, and the Rt. Hon. Sir James Stirling.

Visitors—Dr. J. Mitchell Bruce, Mr. Dugald Clerk, Sir John George Craggs, Mr. H. Cunynghame, Mr. G. F. Deacon, Mr. E. Dent, Rev. J. H. Ellis, Mr. R. K. Gray, Mr. C. E. Groves, Mr. F. G. Henriques, Mr. A. C. Ionides, Mr. C. E. Melchers, Mr. E. R. Merton, Mr. H. Swithinbank, and Mr. G. P. Willoughby.

NOTICES OF BOOKS.

Chemical Analysis, Qualitative and Quantitative. By WILLIAM BRIGGS, LL.D., M.A., B.Sc., F.C.S., and R. W. STEWART, D.Sc. (Lond.). Fifth Impression (Fourth Edition). Revised and Enlarged by H. W. BAUSER, M.A. London: W. B. Clive. 1906.

THE fourth edition of this text-book of chemical analysis gives a good collection of group tests, with clear systematic tables, and a useful set of model analyses. It also contains a sketch of gravimetric and volumetric work, sufficient to cover the syllabus for the London Intermediate Science and Preliminary Scientific Examinations. The directions for the analysis of both simple salts and mixtures appear to be clear and adequate, and the student who works through the book should find no difficulty in passing in chemistry in the above examinations; whether he will have been thought to think is another matter. As a book designed especially for the use of examination candidates it certainly fulfils its purpose.

A Three Years' Course of Practical Chemistry. By GEORGE H. MARTIN, M.A., F.C.S., and ELLIS JONES, M.A. Third Year. London: Rivingtons. 1906.

IN the third part of this course of practical chemistry the properties and reactions of a few of the most important non-metallic elements are thoroughly studied, and useful sets of questions are included, special stress being laid upon chemical arithmetic. The general plan is the same as that adopted in the first and second parts, and it seems to have been equally well worked out, the greater part of the experimental work being done by the students themselves, although there are necessarily a good many experiments which are better suited for demonstration. The pupil who works through this course of chemistry should acquire a very good insight into the elements of the science, and lay a thoroughly sound foundation for further work.

An Introduction to Chemical Crystallography. By P. GROTH. Authorised Translation by HUGH MARSHALL, D.Sc., F.R.S. London: Gurney and Jackson. 1906.

THIS short treatise on chemical crystallography gives a very brief review of investigations of the relations between the properties of crystalline substances and their chemical constitution. The author summarises the isolated relationships which have been detected, with the hope of thus leading up to the discovery of some general law, and, without going very deeply into the various views which have been put forward from time to time, gives a very concise and accurate statement of the most important aspects of the subject. The translation has been very carefully done, the original being closely followed, while, for the benefit of English students who cannot read German or get access to the original papers, some additional

references have been given to abstracts or translations of them in English.

Grundriss der Elektrochemie. ("Outlines of Electrochemistry"). By DR. HANS JAHN. Second Edition. Wien: Alfred Hölder. 1905.

THE second edition of this text-book of electrochemistry has been almost entirely re-written by the author, but its general character remains unaltered, and the changes in it relate chiefly to work which has been done in the last ten years. The views of Arrhenius are adopted as the basis of the book, and the treatment is mainly mathematical, the author believing that a thorough knowledge of the calculus and skill in using them to be absolutely indispensable to the chemist nowadays. He also lays stress upon the importance of physical methods especially in electrochemistry, and he endeavours to overcome the distrust which chemists sometimes exhibit towards such methods. Some parts of the book are exceedingly difficult to read, and it is hardly one that could advantageously be used by the average student, though it gives a very full exposition of the mathematical theory of electrochemistry.

La Double Réfraction Accidentelle dans les Liquides. ("Accidental Double Refraction in Liquids"). By G. DE METZ. Paris: Gauthier-Villars. 1906.

THIS monograph gives a very good summary of the actual state of our knowledge of the phenomenon of double refraction in liquids. The author discusses the means of producing this phenomenon, and describes the most important parts of his own experimental work, besides that of Kerr and Maxwell; the chapters on theoretical views are perhaps a trifle obscure in some respects, but on the whole a difficult subject is skilfully treated, and in the discussion of the constitution of colloids Bütchli's work is excellently reviewed and illustrated. The account of the discovery of Kerr's phenomenon is very clear, and the question of its identity with the phenomenon of double refraction artificially produced mechanically is lucidly treated. The author shows discrimination in the discussion of the work of different physicists, and without descending to minute detail, he manages to convey to the reader a clear idea of the main aspects of this branch of electric optics, and those who are working on the subject will find much in the book to suggest fresh experiments and new ways of attacking the problem, which may lead to the discovery of the real nature of liquids. The author has for some time devoted himself to the study of the question, and has arrived at some interesting results which are here given in full.

Analyse des Métaux par Electrolyse. ("The Analysis of Metals by Electrolysis"). By A. HOLLARD and L. BERTIAUX. Paris: H. Dunod and E. Pinat. 1906.

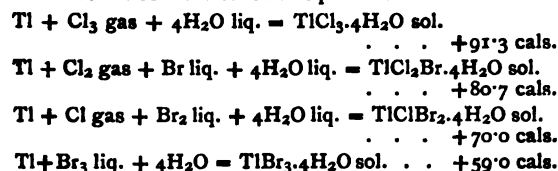
THE special object of the authors of this book has been the description of electrolytic methods of determination in those cases in which gravimetric or volumetric processes are inconvenient or wanting in accuracy; for this purpose they have analysed many mixtures of known proportions, and are thus enabled to report definitely on the degree of accuracy that may reasonably be expected in the use of electrolysis. This alone gives a value to the book, which also contains full practical details of the various methods of separation and estimation, and a scheme of classification of the metals for analytical purposes. One section is devoted to an account of general methods of analysing ores, minerals, alloys, and impure metals. Although an attempt is made to provide a general explanation of the complex phenomena of electrolysis as applied to analysis, the work is chiefly of a practical nature, and, generally speaking, the information given seems to be sufficiently full, while the alloys and minerals treated of have been carefully selected so that they represent the most typical and important of such substances.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances de l'Académie des Sciences. Vol. cxlii., No. 14, April 2, 1906.

Halogen Compounds of Thallium.—V. Thomas.—The author makes a series of researches on the thermochemistry of the halogen compounds of thallium and on the chloruration of thallous chloride. The numbers obtained confirm the chemical existence of the thallic chlorobromides. From the series of equations—



it is seen that the successive replacing of the bromine atoms of the bromide by chlorine atoms evolve practically the same quantity of heat; the first evolving 11.0 cal., the second 10.7 cal., and the third 10.6 cal. When chloruration takes place with excess of chlorine, the process stops when all the thallous chloride is transformed into Ti_2Cl_3 . If chloruration takes place at ordinary temperatures, the bichloride, Ti_2Cl_4 , is always formed. It is necessary, however, to avoid any trace of moisture, as the bichloride is very hygroscopic, and when hydrated rapidly absorbs more chlorine, forming the hydrated thallic chloride. At a high temperature complete chloruration never takes place. If a sealed tube is used under a pressure of six or seven atmospheres, small quantities of the anhydrous chloride are formed in the form of a white sublimate.

No. 15, April 9, 1906.

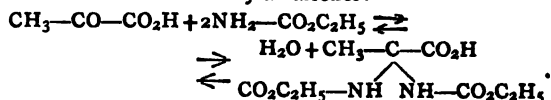
Pyrophosphoric Compounds.—J. Cavalier.—The recognised general formula for pyrophosphoric acid is $\text{P}_2\text{O}_7\text{H}_4$, which is a tetrabasic acid. There exist four sets of salts, the majority of metallic pyrophosphates having the formulæ $\text{P}_2\text{O}_7\text{M}_2\text{H}_2$ and $\text{P}_2\text{O}_7\text{M}_4$, the mono- and tri-metallic salts being rarer and less definite. The existence of the latter, however, has been proved. The author now prepares the neutral pyrophosphates of ethylic, normal propylic, isopropylic, normal butylic, amyllic, and allylic alcohols. The pyrophosphoric ethers are liquid, with an odour resembling the corresponding ortho-phosphates. They decompose with heat, and can be distilled; they are soluble in benzene, carbon disulphide, carbon tetrachloride, and ether. The ethylic compound is soluble in water, the liquid formed being strongly acid.

Barium Iodo-mercurates.—A. Duboin.—The author prepares a saturated solution of barium iodo-mercurate by the same method as that used to prepare calcium and strontium iodo-mercurates. On analysis its composition is found to be—Ba, 12.14 per cent; mercury, 23.32 per cent; iodine, 52.05 per cent; from which the formula $\text{BaI}_2 \cdot 1.33\text{HgI}_2 \cdot 7.76\text{H}_2\text{O}$ can be deduced. This is evidently a mixture of two iodides soluble in alcohol at 96°, the saturated solution having for its density 2.76 at 23.5°. The author also obtains another definite hydrate at -9°C., having the composition $\text{BaI}_2 \cdot 1.30\text{HgI}_2 \cdot 10.41\text{H}_2\text{O}$.

Pure Ferro-molybdenums.—Em. Vigouroux.—The author obtains a series of pure ferro-molybdenums, containing about 80 per cent of molybdenum, by the direct union of iron and molybdenum. These ferro-molybdenums contain four bodies corresponding to definite formulæ. Fe_2Mo should constitute the lowest definite compound capable of being produced in synthesised ferro-molybdenums. This can be obtained from an ingot containing 12.5 per cent Mo

and sufficient iron to form Fe_{12}Mo . Hydrochloric acid dissolves away the iron only, without a trace of molybdenum, and the action only ceases when the residue contains 46.2 per cent of molybdenum.

Influence of the Juxtaposition of the Ketonic and Acid Functions in the same Molecule.—L. J. Simon.—The author showed that the action of pyruvic acid on urethane is an equilibrium action comparable with the etherification of an acid by an alcohol:—



The diurethane pyruvic acid, insoluble in water, slowly disappears by decomposition, and, inversely, the evaporation of the water in a vacuum or on a water-bath causes the initial acid to be reformed. The author investigates the reactions when other solvents, such as alcohol, are used.

Condensation of Acetylenic Amides with Phenols. General Method of Synthesis of β -Oxyphenol Ethylenic Amides.—Ch. Moureu and J. Lazennec.—The acetylenic nitriles can be condensed with alcohols and phenols with formation of addition products resulting from the fixation of the alcoholic or phenolic molecules on to the acetylenic liaison. The acetylenic amides, $\text{R—C}\equiv\text{C—CONH}_2$, are susceptible of giving products of analogous condensation. The authors also describe their experiments with phenols which give well-defined bodies; their composition can be exactly determined by an examination of their hydrolysis products. Under the influence of hot 10 per cent sulphuric acid they are completely decomposed at the end of several hours' boiling with reflux condenser into the corresponding acetone and phenol.

MISCELLANEOUS.

Radio-Activity.—Messrs. Archibald Constable and Co. will publish very shortly a new book by Professor Harry C. Jones, of the Johns Hopkins University, entitled "The Electrical Theory of Matter and Radio-Activity." Professor Jones reviews the whole subject, and brings into line the latest results obtained.

The Society of Dyers and Colourists (London Section).—The Second Annual Dinner of the London Section of the Society of Dyers and Colourists will be held at the Criterion Restaurant, Piccadilly Circus, W., on Wednesday, May 23rd, at 7.30 p.m. Those interested in dyeing and the allied industries who are not members of the Society are specially invited. Tickets 5s. each, and particulars may be obtained from members of the committee, or the Hon. Secretary, Wallace Burton, 219, Shooter's Hill Road, Blackheath, S.E.

Royal Institution.—A General Monthly Meeting of the Members of the Royal Institution was held on the 7th inst., His Grace the Duke of Northumberland, K.G., President, in the chair. Mr. H. Ballantyne, Sir Walter Balfour Barttelot, Bart., Dr. Gustav Hamel, Mr. W. M. Mordey, and Captain Adrian Rose were elected members. The special thanks of the members were returned to Professor J. A. Fleming, F.R.S., for his donation of £26 5s. to the fund for the promotion of Experimental Research at Low Temperatures. It was announced that His Grace the President had nominated the following Vice-Presidents for the ensuing year:—The Right Hon. Lord Alverstone, G.C.M.G., Sir William Huggins, O.M., K.C.B., The Right Hon. Lord Kelvin, O.M., G.C.V.O., Dr. Ludwig Mond, The Right Hon. Lord Sanderson, G.C.B., K.C.M.G., The Right Hon. Sir James Stirling, Sir James Crichton-Brown (Treasurer), and Sir William Crookes (Honorary Secretary).

The International Association for Testing Materials.—It is arranged that this Association, which holds its congresses about every three years in industrial centres in various countries, shall this year meet in the Academy of Science at Brussels, from September 3 to 8. The King of Belgium accords the Congress his patronage, while Prince Albert of Belgium will be one of the honorary presidents, as also will the Ministers of Finance, Railways, War, and Trade, and the Mayor of Brussels. The programme of excursions includes visits to the Harbour Works at Brussels and at Antwerp, the Steel Works of the John Cockerill Company at Seraing, the Arsenal at Mechlin, and the Harbour Works at Zeebrugge. Among the papers to be read will be one on the Industries of Belgium, by Baron E. de Laveleye and M. Camerman. It is expected that a considerable number of members and delegates from this country will be present at the Congress. Mr. J. E. Stead, F.R.S., Middlesborough, is the English Secretary of this institution.

Institute of Chemistry of Great Britain and Ireland.—*Pass List of the April Examinations.*—Of seven candidates who entered for the intermediate examinations, six passed: C. T. Bennett, B.Sc. (Lond.); S. H. Groenewoud; B. R. James; G. R. W. Lawson; E. H. Merritt, B.Sc. (Lond.); and C. A. Wotham, B.Sc. (Lond.). In the final examination for the Associateship (A.I.C.), of four examined in the branch of Mineral Chemistry, three passed: G. N. Bacon, Assoc. R.C.Sc. (Lond.), B.Sc. (Lond.); F. M. G. Johnson, M.Sc. (Montreal); and E. J. Wilson, B.A. (Cantab.). Of four in the branch of Metallurgical Chemistry, one passed: H. J. Bailey. Two candidates presented themselves in Organic Chemistry, and both passed: L. H. Berry, and O. W. Stickland, B.Sc. (Lond.). Of seven who entered in the branch of the Analysis of Food and Drugs and of Water, including an examination in Therapeutics, Pharmacology, and Microscopy, the following five passed: B. J. Eaton, W. R. Mummery, F. L. Okell, R. P. Page, and R. G. Pelly. The examiners in chemistry were Mr. W. W. Fisher, M.A., F.I.C., and Dr. G. G. Henderson, M.A., F.I.C. The examination in Therapeutics, Pharmacology, and Microscopy was conducted by Dr. F. Gowland Hopkins, M.A., F.R.S., F.I.C.

Society of Dyers and Colourists.—*Prizes for the Solution of Technical Problems:*—

1. Prize of £20 for a full investigation of the average degree of tendering brought about in cotton-yarn of various qualities by—(a) Cross dyeing with acid colours, and (b) dyeing aniline black, with the object of fixing standards for the trade.
2. Prize of £10 for a practical method of so treating or preparing cotton-yarn as to cause it to resist direct dyeing cotton colours. (The object desired is the production of a pattern or mixed effect in the piece dyeing of all cotton goods).
3. Prize of £10 for a practical method of dyeing full shades of basic colours on cotton, fast to rubbing.
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NOTES AND QUERIES.

*. Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Estimation of Boron.—Would one of your readers supply a reference where I can find methods of determining boron in alloys with iron and manganese, and also of estimating boric acid by means of standard alkaline solutions? Also, what indicators are applicable for this purpose?—A. A.

MEETINGS FOR THE WEEK.

MONDAY, 14th.—Society of Arts, 8. (Cantor Lectures). "Heraldry in relation to the Applied Arts," by George W. Eve.

TUESDAY, 15th.—Royal Institution, 5. "Glands and their Products," by Prof. William Stirling, M.D., &c.
Faraday Society, 8. "The Electrolysis of Fused Zinc Chloride in Cells Heated Externally," by Julius L. F. Vogel. "Sensitiveness of the Platinum Electrode," by H. D. Law.

WEDNESDAY, 16th.—Society of Arts, 8. "The Development of Water-marking in Hand-made and Machine-made Papers," by Clayton Beadle.
Microscopical, 8. Exhibition of Pond Life.

THURSDAY, 17th.—Royal Institution, 5. "The Influence of Ptolemaic Egypt on Græco-Roman Civilisation," by the Rev. J. P. Mahaffy, C.V.O., D.D., &c.
Chemical, 8.30. "Relation between Absorption Spectra and Chemical Constitution—Part VI, The Phenylhydrazones of Simple Aldehydes and Ketones," by E. C. C. Baly and W. S. Tuck. "Aromatic Compounds obtained from the Hydroaromatic Series—Part II., Action of Phosphorus Pentachloride on Trimethyl-dihydroresorcin," by A. W. Crossley and J. S. Hink. "Studies of Dynamic Isomerism—Part V., Isomeric Sulphonic Derivatives of Camphor," by T. M. Lowry and E. H. Magon. "Studies on Basic Carbonates—Part I., Magnesium Carbonates," by W. A. Davis.

FRIDAY, 18th.—Royal Institution, 9. "International Science," by Prof. Arthur Schuster, F.R.S., &c.

SATURDAY, 19th.—Royal Institution, 3. "The Old and New Chemistry," by Prof. Sir James Dewar, D.Sc., F.R.S., &c.

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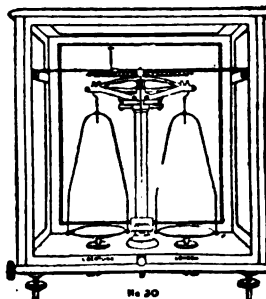
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VOL. XCIII., No. 2425.

BACK REACTIONS IN IODINE TITRATIONS.

By JOHN HUGHES DAVIES and EDGAR PHILLIP PERMAN.

It is often noticed when making quantitative determinations involving the liberation of iodine from potassium iodide and the titration of the liberated iodine by sodium thiosulphate or arsenious acid solution, using starch as an indicator, that the end-point of the reaction is uncertain owing to the reappearance of the blue colour on standing a short time. We are not aware that any explanation of this phenomenon has been offered, and have now made some experiments in order to throw light on the subject.

We find that the appearance or otherwise of the "back reaction" depends on the concentration of the potassium iodide in solution. The "back reaction" is most marked in those cases in which the iodine is liberated by copper sulphate or potassium dichromate, but takes place only to a very slight extent when bleaching-powder or permanganate is used.

The explanation of the reappearance of the blue colour lies evidently in the comparative slowness (and incompleteness) of the liberation of iodine from potassium iodide, in dilute solution, by the substances mentioned. Sufficient increase of concentration of the iodide solution makes the reaction very nearly instantaneous, and thus increases the accuracy of the titration by avoiding any possible "back reaction." It was found that suitable quantities of potassium iodide were 1 grm. in 10 c.c. water for 25 c.c. $N/10$ $K_2Cr_2O_7$ solution, and 2 grms. in 20 c.c. water for 1 grm. $CuSO_4 \cdot 5H_2O$ in 50 c.c. water.

University College, Cardiff.

NOTE ON THE SAMPLING OF GOLD ALLOYS.

By ERNEST A. SMITH, A.R.S.M.

In assaying the various gold alloys used by jewellers and goldsmiths, great care has to be exercised in the selection of the sample, as the surface is usually richer in gold than the interior, in consequence of the pickling or blanching to which these alloys, whether in the form of sheet metal, wire, or gold wares, are invariably subjected.

During the process of manufacture the alloys, which consist of gold alloyed with copper and silver in various proportions, require to be repeatedly heated to a red-heat for annealing and for soldering purposes, with the result that the copper of the surface is oxidised more or less deeply.

To remove the coating of oxide thus formed the alloys are dipped for a short time in hot dilute sulphuric acid or nitric acid, which dissolves the copper oxide and leaves a film of more or less pure gold.

It is evident that these operations, repeated several times on the same piece, produce a refining of the surface, the fineness of which is notably different from the rest of the mass. Consequently, one of the first precautions to be taken when sampling these alloys for assaying is to remove this enriched or coloured surface, as it is obvious that if it is included in the sample the result obtained will not represent the true fineness of the alloy but will be above it. The thickness of the film of gold resulting from the pickling necessarily varies considerably, and is chiefly dependent on the composition of the alloy, the strength and nature of the acid used, and the length of time the alloy is allowed to remain in the acid.

It may be pointed out that finished articles of jewellery and of goldsmiths' work are very frequently coloured by a

special process, the film of gold in this case being usually thicker than that which results from the ordinary pickling process; but gold articles when submitted for assay are invariably in an unfinished condition, and have only been subjected to immersion in dilute acid to clean them.

No figures appear to have been published hitherto to show to what extent the accuracy of the gold assay may be affected by including the coloured surface in the assay sample; the following results therefore, based on several years' experience with these alloys, may not be without interest.

The gold alloys received from bullion dealers are usually in the form of thin sheet or wire, and can, as a general rule, be readily sampled by cutting off a piece with the shears or pliers; but in the case of gold wares and jewellery, owing to the nature of the articles, it is very seldom that a cutting sample can be obtained without risk of injuring the article, and careful scraping of the surface has to be resorted to. A scraping sample, however, is not so satisfactory, and it is advisable to take a cutting sample whenever possible, as it will more truly represent the mass of the metal than a sample scraped from the surface.

It is hardly necessary to say that when the coloured surface is included in the sample the errors in the gold assay are much greater with a scraping sample than with a cutting sample, as the former will contain a larger proportion of the enriched surface.

Experience has shown that, under similar conditions of pickling, the errors are usually greater with alloys in which the gold is alloyed mainly or entirely with copper than with alloys in which the copper is partly or entirely replaced by silver, as the copper is more readily acted upon by the acid during pickling.

The figures given in Table I. show to what extent the results of the gold assay are influenced by the coloured surface in the case of samples cut from sheet metal. Samples of sheet metal of different gold standards were carefully cleaned and rolled to various gauges, then simultaneously heated to a dull red heat, and pickled in hot dilute nitric acid for two minutes to ensure uniformity of treatment.

When removed from the acid the strips were rinsed in distilled water and dried. To ascertain the true fineness of the alloys, separate assays were made on samples from which the coloured surface had been carefully removed.

The assays were made in the ordinary way, check assays being added in all cases.

TABLE I.—Assays of Cutting Samples, including Coloured Surface.

	Thickness of sheet		Description of sample	Gold per 1000 parts.	Difference per 1000 parts.
	B.M.G.	Inches.			
Sample A, 9 Carat Gold Sheet.					
1.	11	0.032	Colour removed	375.0	—
2.	11	0.032	Colour included	380.6	+5.6
3.	8	0.021	" "	380.8	+5.8
4.	4	0.012	" "	381.3	+6.3
5.	1	0.008	" "	381.5	+6.5
Sample B, 9 Carat Gold Sheet.					
1.	8	0.021	Colour removed	376.6	—
2.	8	0.021	Colour included	382.0	+5.4
3.	4	0.012	" "	384.6	+8.0
4.	1	0.008	" "	389.3	+12.7
12 Carat Gold Sheet.					
1.	8	0.021	Colour removed	500.1	—
2.	8	0.021	Colour included	500.3	+0.2
3.	4	0.012	" "	500.4	+0.3
4.	1	0.008	" "	500.8	+0.7
15 Carat Gold Sheet.					
1.	8	0.021	Colour removed	631.2	—
2.	8	0.021	Colour included	632.0	+0.8
3.	4	0.012	" "	632.1	+0.9
4.	1	0.008	" "	632.5	+1.3

As previously stated, the duration of the pickling has an important bearing on the assay result, and with commercial samples it is not an uncommon thing to get greater differences than those given in the table, when the colour is not removed.

As a general rule, the errors are smaller with the alloys of higher standard, as there is less base metal to be dissolved out.

With samples cut from the sheet the thickness of the metal necessarily plays an important part, the errors resulting from the coloured surface being minimised with an increase in the thickness of the sheet. Metal sheet gauged to the higher gauges given in the table is that most frequently met with in goldsmiths' work, but metal rolled to the lower gauges is also in use.

In the year 1900, it was decided by the wardens of the various assay offices that "in future no hollow gold wares would be assayed and hall-marked if made of a thinner gauge than No. 36 of the legal standard wire gauge, the equivalent of which is 0.0076 inch" (*Thirty-third Mint Report*, 1902, p. 67), or slightly thinner than No. 1. of the Birmingham Metal Gauge, by which sheet metal is invariably gauged.

It may be pointed out, however, that the decision applies only to *hollow* gold wares, and as the authorities at the assay offices have no jurisdiction over the manufacture of jewellery it not infrequently happens that articles made from very thin sheet are submitted for assay.

The errors introduced by including the coloured surface in a scraping sample are given in Table II.

The first sample in each case was obtained by carefully scraping the surface until the natural colour of the alloy was reached; the second sample was then scraped from the same place.

As the first scraping removes not only the outer layer but more or less of the original alloy, it necessarily follows that the assay results will vary in accordance with the depth to which the scraping has been carried. In the case of articles made from very thin sheet, a large surface has to be scraped in order to secure a quantity sufficient for assay, and as the sample under these conditions will consist very largely of the coloured surface, the errors on the gold assay are necessarily very considerable.

This fact accounts for some of the very high results given in the table.

A larger number of results have been given for 9 carat gold than for any other standard, this alloy being the one most frequently received for assay, more than 50 per cent of the wares passing through the Government Assay Offices for hall-marking being made from gold of this standard.

TABLE II.—Assays of Scraping Samples, including Coloured Surface.

Sample.	Gold per 1000 parts.		Difference due to coloured surface. Per 1000 parts.
	First scraping (including colour).	Second scraping (correct fineness of alloy).	
9 Carat Gold.			
A.	392.8	382.0	+ 10.8
B.	489.0	392.0	+ 97.0
C.	424.0	376.3	+ 47.7
D.	514.5	378.8	+ 135.7
E.	440.0	382.0	+ 58.0
15 Carat Gold.			
A.	706.8	630.2	+ 76.6
B.	703.5	630.0	+ 73.5
C.	688.7	632.4	+ 56.3
18 Carat Gold.			
A.	772.6	755.7	+ 16.9

In taking scraping samples, after removing the coloured surface, it is very important to scrape as deeply as the nature of the article will permit, especially with articles which have been subjected to any stamping operations in

the course of manufacture; this will be evident from the following example, which is by no means an isolated case.

Three scraping samples were taken from a sheet of 15 carat gold of about 18 B.M.G. which had been repeatedly struck between steel dies, and assayed for gold with the following results:—

	Gold per 1000 parts.
First scraping (including coloured surface) ..	633.0
Second scraping	623.7
Third scraping	625.0

As the second assay gave a result a little below the legal 15 carat standard, viz., 625.0 per 1000, the third scraping sample was taken and assayed with the result quoted. From these results it would appear that the oxide resulting from the several annealings was only removed by pickling after the final annealing and not after each annealing, with the result that the oxide was driven into the metal when it was stamped between the dies, giving rise to the low result of the second sample. To confirm the result of the third scraping sample, a piece of metal was cut from the sheet, and the coloured surface removed. When assayed it was found to be 625.0 fine, thus proving that the third scraping sample truly represented the bulk of the alloy.

The results given in the tables prove the impossibility of getting a correct result for the gold content of an alloy when the external layer is introduced into the sample, whether taken by cutting or by scraping, and they also emphasize the importance of carefully removing the coloured surface from gold alloys of all standards before taking the sample for assay, particularly when scraping samples have to be taken.

A SYSTEM OF QUALITATIVE ANALYSIS INCLUDING NEARLY ALL THE METALLIC ELEMENTS.*

PART II.—ANALYSIS OF THE TUNGSTEN GROUP.

By ARTHUR A. NOYES.

(Continued from p. 218).

Test Analyses (continued).

Tantalum.—This element was present in quantity of 2 m.grms. in fourteen of the mixtures analysed, of 3 m.grms. in two of them, and of 1 m.grm. in one of them. A satisfactory test for it was obtained in every case, except one (T. A. 3) in which 300 m.grms. of titanium and 300 m.grms. of tin were simultaneously present, and it was found, even in this mixture, in a second analysis (T. A. 5). Its detection succeeded repeatedly in the presence of 300 m.grms. of titanium, or in that of 300 m.grms. tungsten, or in that of 300 m.grms. tin. It succeeded also in the cases where 400—500 m.grms. molybdenum (T. A. 16, 37), or 100 m.grms. niobium (T. A. 15), or 400 m.grms. antimony (T. A. 36) were present. Two m.grms. can therefore always be detected.

The "blank" tests for tantalum (that is, those in the absence of this element) were somewhat inconclusive in those mixtures containing much titanium (T. A. 9, 11, 32, 35), owing to the presence of a minute quantity of tantalum in the titanium sample used; but the precipitates were always very small (1—2 m.grms.), and there is little doubt that with pure material perfect blanks would be obtained. In two cases (T. A. 31, 32), also, a light flocculent precipitate separated in place of the heavy viscous one characteristic of tantalum. Good blanks were obtained in the other cases (T. A. 2, 28, 34, 38). While a further study of the blanks obtaining in testing for this element according to the procedure is desirable, especially since no good confirmatory test for it is known, yet it is hardly possible that error will be made, even with respect to small quantities, by any one who first makes himself familiar

* From the *Technology Quarterly*, xvii., No. 3.

with the appearance of the potassium fluoxytantalate and fluotitanite.

Niobium.—There would be no difficulty whatever in detecting 2 m.grms., or even less, of this element, provided the first test with mercuric chloride could be relied upon; but owing to the possible presence of tungsten or molybdenum, it is ordinarily necessary to fuse with sodium carbonate and sulphur, and then apply a second test. The results show that under these circumstances 2 m.grms. is near the limit of detection. This quantity was present in nine of the mixtures analysed; and the second test failed in five, and succeeded in three of them (it being omitted in one case). Three m.grms. were present in two mixtures and 5 m.grms. in two others, and in all of these the test was satisfactory. It succeeded with 2 m.grms. in the presence of 400 m.grms. of antimony (T. A. 31), or of 200 m.grms. of titanium (T. A. 12), or of 100 m.grms. tantalum (T. A. 14), and with 5 m.grms. in the presence of 300 m.grms. of tin (T. A. 11, 19). It seems safe to conclude that 3 m.grms. can be detected under almost any conditions.

The question of blank tests for this element in the presence of much titanium, tungsten, or molybdenum is an especially important one, and many mixtures were prepared and analysed with reference to it. A perfectly clear solution, or a slighter turbidity than would have resulted from 1 m.grm. of niobium, was obtained in the second mercuric chloride test from nine mixtures containing 300–500 m.grms. of titanium. In three of these mixtures 300–400 m.grms., and in two of them 4–5 m.grms. tungsten were simultaneously present. The result, too, was satisfactory in the four cases where tungsten was present with only a small quantity of titanium. This was also true where the mixtures contained much tin, either with or without much tungsten. In two of the five mixtures (T. A. 16, 35) in which 300–500 m.grms. of molybdenum were present, the second mercuric chloride test showed a decided turbidity, while in the other three (T. A. 17, 31, 39) a good blank was obtained; but in all these analyses except the last (T. A. 39) the ammonium sulphide precipitate was not re-digested with ammonia—a precaution that was adopted only near the end of the work. The results of the blank tests for niobium may therefore be considered satisfactory under all circumstances, provided regard is paid to all the precautions described in the procedure.

Titanium.—The results for this element require little discussion. The yellow or orange-red colour produced with hydrogen peroxide constitutes so delicate a test that in every mixture containing 1 or 2 m.grms. of it, it was conclusively detected. The test is so characteristic that blank tests were considered unnecessary, especially since its presence is ordinarily confirmed by the reddish violet colour produced with zinc.

Tin.—The only analyses in which the final procedure for the separation and detection of tin was employed are those of the third and fourth series; but Test Analyses, 1, 2, and 8 also deserve consideration, since the omission of the ignition of the sulphide could hardly affect the tin test. The results show that 2 m.grms. escape detection when 400 m.grms. of either titanium (T. A. 32), antimony (T. A. 31), or molybdenum (T. A. 34) are present, and that this is true of even 4 m.grms. in the presence of 400 m.grms. of molybdenum (T. A. 37), or in the simultaneous presence of 400 m.grms. of titanium and an equal quantity of molybdenum (T. A. 35) or tungsten (T. A. 33). On the other hand, the test succeeded with 4 m.grms. of tin in presence of 400 m.grms. of antimony (T. A. 36), with 8 m.grms. of tin in the presence of 400 m.grms. of molybdenum (T. A. 44), and with 1 m.grm. of tin in the presence of 300 m.grms. of tungsten (T. A. 42), or of 100 m.grms. of titanium (T. A. 2). It seems therefore fair to set the limit of detection at 3 m.grms. in the presence of much antimony or titanium, at 6 m.grms. in that of much molybdenum, and at 1 m.grm. in that of much tungsten.

Blank analyses were considered unnecessary, since experiments (see C. E., P. 41, N. 1) showed the complete

insolubility in hot hydrochloric acid of the ignited sulphides of the other elements which reduce mercuric chloride.

Tungsten.—In the case of this element, all analyses except those of the first series were made by the final procedure. The analyses of the first series in which the test succeeded (T. A. 2, 7, 22, 23) also deserve consideration, since the omission in them of the ignition of the sulphide could only have the effect of making the test less delicate. It will be seen from the tables that the detection of 2 m.grms. failed in the presence of 300–400 m.grms. of antimony (T. A. 31), or of titanium (T. A. 29), or of 100 m.grms. or more of molybdenum (T. A. 28, 31, 47); and that that of 4 m.grms. failed in the simultaneous presence of 400 m.grms. each of titanium and molybdenum (T. A. 35). On the other hand, 3 m.grms. were detected when no other element was in great excess (T. A. 2, 27) or when much tin was present (T. A. 40, 41); and 4 m.grms. were detected when the mixture also contained 400 m.grms. of titanium (T. A. 32) or of antimony (T. A. 36), or 100 or more m.grms. of molybdenum (T. A. 34, 37, 46). Eight m.grms. were detected in the presence of 20 m.grms. of phosphate (T. A. 43), but a less quantity could probably have been found. The limit of detection may therefore be set at 2 m.grms. when it is present either alone or with a large quantity of tin, and at 3 or 4 m.grms. when a large quantity of antimony, titanium, or molybdenum is simultaneously present.

Molybdenum and tin are the only elements that could give rise to a precipitate in a blank analysis at the point where the ignited sulphides are dissolved in nitric and hydrochloric acid (P. 43); and mistake from this source is entirely guarded against by the subsequent evaporation with sulphuric acid, and by the confirmatory test with zinc.

Antimony, Arsenic, and Molybdenum.—The final detection of these elements is effected in connection with the later groups. It is therefore sufficient to show that even a small quantity of any of them present in the solution of this group is precipitated by hydrogen sulphide from the hydrofluoric acid solution (P. 31). This is shown by the above analyses for 1 or 2 m.grms. of antimony (T. A. 1, 25, 26) and of molybdenum (T. A. 1, 32, 33). Special experiments have confirmed this result, and have shown that 2 m.grms. of arsenic are also precipitated (see C. E., P. 31, N. 3).

Summary.—The preceding discussion may be summarised by the following statements:—Four m.grms. of any element of this group, when present in the hydrofluoric acid solution at the start, can be detected with certainty by the procedure described in this article, even when a quantity as large as 300–400 m.grms. of any other element of the group is simultaneously present. Two m.grms. of titanium, tantalum, antimony, arsenic, or molybdenum, or three of niobium, can be detected with certainty in any combination whatever with other elements. Two m.grms. of tin or tungsten can each be detected alone or in the presence of a very large quantity of the other, but not in the presence of a large quantity of the elements precipitated by hydrogen sulphide or ammonium sulphide. Eight m.grms. of tungsten, and probably much less, can be detected in the presence of a considerable quantity of phosphate. There is no possibility of concluding that any absent element is present as a result of a failure of the final test to yield a blank, except in the cases of tantalum and niobium; and observance of all the precautions described and of the remarks as to the interpretation of the results will prevent any mistake in these cases.

In conclusion, it need hardly be said that skill in the manipulation of exact qualitative analysis and some slight experience with this process itself are essential in order to secure the degree of delicacy just referred to it. But the attempt has been made to make the directions so detailed and explicit that it is believed that an analyst possessing such skill will be able to approximate closely to these results after working through a single mixture containing small quantities of all the elements of the group.

(To be continued).

AN IMPROVED EXTRACTION CUP.

By E. BRUCE WARREN.

THIS cup has been designed for general extraction purposes, the novel feature being that the substance under treatment whilst being acted upon by the pure solvent is kept at the



FIG. 1.

temperature of the boiling-point of the solvent employed, thus greatly facilitating the extraction of difficultly soluble substances. Figs. 1 and 2 show two designs of this cup.



FIG. 2.

In Fig. 1 the cup has a discharging pipe from the bottom, which is carried upwards to a point below the top

of the cup. This form of cup enables the residue to be dried in a stream of any gas if the substance is likely to undergo any change during drying in the ordinary way.

In Fig. 2 the cup containing the substance under examination is supported in a tube, and the solvent is kept in the cup at a height corresponding to the height of the outer tube.

In use, the bottom of the cup is covered by a plug of wool or other suitable substance and the material under examination is weighed into it. The cup is placed in a flask, sufficient solvent poured in, and the flask fitted with a reflux condenser arranged to let the condensed solvent fall back into the cup. This percolates through the material and passes away with the soluble matter through the outlet back into the flask. After the extraction is completed, the residue can be dried in a current of gas by connecting up with the outlet of the cup.

We have found this design of extraction apparatus to yield very satisfactory results at the Indiarubber Works at Silvertown, being both efficient and rapid; substances even as difficultly soluble as Carnaüba wax being extracted by acetone in a short time.

This cup is manufactured by Messrs. Müller, Orme, and Co., and is sold by them under the name of the "Flow."

A REVISION OF THE ATOMIC WEIGHTS OF SODIUM AND CHLORINE.*

By THEODORE WILLIAM RICHARDS
and
ROGER CLARK WELLS.

(Continued from p. 219).

THE RATIO OF SODIC TO ARGENTIC CHLORIDE.

Two obvious means are available for determining the combining weight of sodic chloride—one, by weighing the amount of argentic chloride which may be made from a given weight of this salt; the other, by discovering the weight of silver needed to precipitate all its chlorine. Both these methods involve a consideration of all the special points mentioned in the preceding sections, and both are essentially gravimetric, although both involve the use of the nephelometer in order to estimate the last traces of silver or chlorine in the supernatant liquid.

The actual investigation of these two methods proceeded simultaneously in order to economise the time during the long delays to which each was frequently subject. Indeed, in some of the previous work in this laboratory, where the pure materials were scarce, the two methods were actually combined in the same analysis, by first determining the weight of silver needed to precipitate the chloride, and then weighing the chloride produced. This last procedure is, nevertheless, somewhat objectionable on account of its complication. It is, moreover, subject to slight opposing errors; the avoidance of one of these involves the admission of the other, as has already been pointed out in a preceding communication (Richards and Archibald on the Atomic Weight of Cæsium, *Proc. Amer. Acad.*, 1903, xxxviii., 443). The magnitude of the uncertainty thus caused is not great, and the final error involved is insignificant even in atomic weight investigation of ordinary calibre, usually not exceeding 0.01 per cent; but in the present case extraordinary accuracy was sought. Hence, although the methods were studied simultaneously, they were never combined in the same analysis. For this reason the work is easily divided into two sections, each treating one of the two ratios named above. Of these it is convenient to discuss first the ratio of the chlorides to one another; and this discussion is taken up in the present chapter.

After the preliminary experiments had indicated the nature of the problems involved, a single specimen of pure

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sodic chloride was used for analysis until all the details of analysis were perfected. Then the various samples of salt were analysed by methods of known precision. The temptation to vary two conditions in a single new experiment is thus easily avoided. The detailed description of every step of the analytical process follows.

It was soon found that salt, when free from excess of acid, can be fused in a platinum crucible in the air without either attacking the crucible to an important extent, or becoming appreciably alkaline. Quantitatively, it was later proved, as will be shown, that salt thus prepared is identical to that fused in a platinum boat in a vacuum and transferred and weighed in the familiar "bottling apparatus" which has served so conveniently for hygroscopic substances. It was found, moreover, that a crucible full of salt thus prepared did not gain in weight during the operation of weighing, on account of the dry winter atmosphere of the steam-heated weighing-room. Probably this would not have been the case in a warm climate; but being the case, it facilitated greatly the present work.

The crucible generally used for fusing the salt was, of course, previously freed from iron, and during twenty-three fusions it lost only 0.47 m.grm., or 0.02 m.grm. each time. It is possible that a portion of the weight was lost by volatilisation during the preliminary ignitions of the crucible when it was being prepared for weighing alone; but probably most of this trace of platinum went into solution in the salt. The weight of the crucible before fusion was always taken as the true one, however; therefore even this slight possibility of loss of course could not affect appreciably the quantitative result.

During the fusion of the salt the crucible was enclosed in a larger covered Berlin porcelain crucible, especially made to fit it, and heated by a blast-lamp in a small clay furnace. The products of combustion were suitably deflected, so that only pure air should be in contact with the salt in the crucible. The fused salt was cooled in a desiccator in the balance room, and the weighing was conducted by the substitution of a suitable tare-crucible, as usual.

In order to avoid all risk of accidental loss, the salt was dissolved from the crucible by placing it in a large covered beaker, and after covering it carefully with water, agitating it gently from time to time until solution was complete. This solution, thus carefully prepared, was transferred to a 2-litre Erlenmeyer flask with a well-ground glass stopper, where it was diluted to suitable volume (over 5 litres per mol.). Here it was precipitated by the at least equally dilute argentic nitrate in excess. The precipitate was freed from the supernatant mother-liquor by filtration as soon as it had cleared enough to make this separation possible. Promptness is needed in order to avoid the permanent occlusion of sodic and argentic nitrate in the manner already stated, but too great promptness prevents complete precipitation of the silver halide. About fifteen hours are usually allowed for the settling of the solution. Agitation is not necessary.

The clear supernatant liquid was decanted through a large carefully prepared Gooch crucible. The mat of this Gooch filter, through which all the mother-liquor and wash-waters were passed, was prepared of selected fibrous asbestos shreds, which had been boiled with nitric acid and well washed. The mat was a trifle thicker than a filter-paper—so that the holes of the crucible were indistinctly visible when viewed in front of a strong light—and was not allowed to extend up the sides of the crucible. A preliminary ignition of the mat was found to result in a slight disintegration and brown coloration, and caused loss during washing; therefore before weighing the mat was dried at a lower temperature, namely, about 150°. This same temperature was used later in drying the argentic chloride. During the operation of washing, at first all but a trace of the precipitate was left in the flask, and it was then re-washed by decantation. After each washing, the precipitate was allowed to drain several minutes, for silver chloride is known to hold water like a sponge. At the

second or third washing a disintegration of the precipitate often took place, resulting in an emulsion-like mixture, but in most cases this could be prevented by the presence of a little nitric acid. The precipitation had been made with silver nitrate in excess. The first three washings, of 0.2 litre each, were made with very dilute silver nitrate, and this was displaced in the fourth washing by about 0.05 litre of pure water. In such wash waters, which, with the mother-liquor, usually amounted to about 2 litres, no trace of chloride could be found by the nephelometer. The main weight of silver chloride was corrected by + 0.06 m.grm., however, in every case, for the otherwise determined solubility in such wash waters (see p. 203). From five to eight subsequent treatments with pure water, very slightly acid, in 0.1 litre portions, completed the washing. These later wash waters were treated in the manner described below, in order to determine the chloride which they always contained.

The precipitate was now transferred to the filtering crucible wholly by means of a stream of water. It was found convenient first to transfer and pack down by air pressure a small portion of the precipitate on the asbestos mat, in order that the greater part of the argentic chloride might be easily separated from this mat for fusion. By resting the lip of the flask on the Gooch crucible it is possible to direct a stream of water from a wash-bottle into the flask and finally wash out every particle of precipitate. In the most careful experiments the wash-bottle was provided with protecting bulbs to preserve the purity of the water, or was dispensed with altogether, being replaced by a jet driven by hydrostatic pressure. At this stage the cloth covering was necessarily removed from the flask. The flask was finally rinsed with ammonia, which was carefully tested for a possible trace of silver chloride.

On drying the precipitate the temperature was made to rise very slowly above 100° during two or three hours. This deliberate drying was found to be more effective than the immediate application of high temperature, presumably because a hard coating was not then formed about the mass of the precipitate. Finally, the temperature was maintained at about 150° for two or three hours longer. In this way fairly constant weight is easily attained, which fifteen hours' further heating at 150° changes very slightly. The last traces of water were always expelled by fusion, as is described below.

In many of the experiments the silver chloride was dried in an electric drying oven heated by hot platinum wires conveying a current of an ampere or two (Richards, *Am. Chem. Journ.*, 1899, xxii., 45). When properly constructed this oven gives very satisfactory results. The working of the oven is somewhat more satisfactory if the platinum wires are not heated to redness. In some of the later experiments the precipitate was dried in a clean copper oven heated by a gas flame with no apparent difference in result.

The precipitate thus collected and dried was carefully weighed and then freed from adhering asbestos and transferred to a weighed porcelain or quartz crucible for fusion. The crucible and contents were tared against a similar crucible and contents, and the argentic chloride under examination was then fused in a clean oven constructed from a large porcelain crucible, from the interior of which the products of combustion of the gas were carefully deflected. The fusion is necessary, because minute drops of water are always held by the hardened argentic chloride in sealed cells. It is true that the loss on fusion is not great, never amounting to 0.01 per cent of the weight of the precipitate, but it was enough to receive consideration. In every case it was determined and the appropriate individual correction applied to each weight. The maximum value of the correction was 0.008 per cent, and the minimum 0.003 per cent. That the loss was not due to volatilisation of silver chloride is easily shown by the invariability of the weight upon fusing a second time. That it was not due to accidental reduction was shown in the later experiments by fusing again in a stream of chlorine, when the mass gained on the average only 0.001 per cent. The fused

argentic chloride, even before its treatment with chlorine, was usually pearly and colourless.

In any case, it is clear that this loss on fusion, which has been rejected as of doubtful significance by some authors (Scott, *Journ. Chem. Soc. Trans.*, 1901, lxxix., 147), really represents included water, and must be heeded.

The determination of the weight of the main mass of the precipitate having been thus determined, attention must be directed to several other precautions necessary to ensure exact work. In the first place, all the liquid which has passed through the Gooch crucible was passed through a small washed filter, in order to collect the minute asbestos shreds which even the best fibrous asbestos seems to lose under pressure. This was done as soon as possible after filtration, and before proceeding to solubility determination. A syphon filtering arrangement, or a large inverted flask fitted with a carefully cleansed rubber stopper with two tubes arranged so as to maintain constant level, obviated the tedious process of filtering by hand. The little filter was finally washed free from salts, and the weight of the ignited residue (minus the filter ash) added to the weight of Gooch crucible and precipitate. This residue usually amounted to only one or two tenths of a m.grm. Observation in the microscope easily showed that the residue was really asbestos.

Because it was the object of the research to test every step of the processes involved, the loss and recovery of asbestos shreds was tested by special blank experiments. Large volumes of water were passed through empty Gooch crucibles provided with the usual mats; and the loss of each crucible after drying was found, as well as the weight of shreds recovered from the filtered water. Below are given the results of these blank experiments:—

	Loss of crucible.	Asbestos found.
1	0.00034	0.00034
2	0.00016	0.00045
3	0.00019	0.00016
Average ..	0.00023	0.00032

In two of these experiments the loss very nearly equalled the amount of asbestos found; the other one must have been contaminated by an accidental outside impurity; but the results of the blank experiments are enough to show that the correction is a real one and not to be omitted.

(To be continued).

Royal Institution.—On Thursday next, May 24, at 5 o'clock, Professor W. J. Sollas begins a course of three lectures at the Royal Institution on "Man and the Glacial Period." The Friday Evening Discourse on May 25 will be delivered by Mr. Leonard Hill on "Compressed Air and its Physiological Effects."

Madame Curie.—We quote the following paragraph from *The Times* of May 14th:—"The Governing Board of the Paris University have decided to maintain the Chair of General Physics held at the time of his death by M. Curie, and to propose the name of his widow as their late colleague's successor. The Minister of Education has signed a decree accepting this proposal. Mme. Curie was the constant collaborator with her husband in the researches which have made their name famous throughout the world. One of the chief promoters of this movement has been the eminent chemist M. Berthelot."

New Apparatus.—Messrs. John J. Griffin and Sons, Ltd., have recently placed upon the market a novel form of pipette, called the "Struthers Syphon Pipette." It is claimed that it is easier to fill than the old straight form, shorter and more compact, and that the adjustment and flow of discharge can be regulated with great care and precision; it also avoids danger when using corrosive liquids, as there is no need to adjust the liquid to the mark by suction. The same firm have also listed the "Dougal Assay Tube," specially designed to replace the assay flask in parting and similar operations.

PROCEEDINGS OF SOCIETIES.

THE ROYAL SOCIETY.

CONVERSAZIONE AT BURLINGTON HOUSE.

ON May 9th the first of the Annual Conversazioni of the Royal Society took place. Among the more important exhibits we note the following:—

Sir OLIVER LODGE, F.R.S., and Dr. ALEXANDER MUIRHEAD, F.R.S. Wireless Telegraphy Apparatus for Military field purposes.

1. A portable pack-transport set of wireless telegraphy apparatus for military field purposes, available for communications across country for distances up to 50 miles, or 150 miles over sea; with electric valves employed to accumulate the impulses of a small coil and battery, or small dynamo, so as to give discharges of energy only otherwise obtainable from a large and heavy source of electric supply. The arrangement needs no earth connection, nor must it have any when it is required to work over long distances with the greatest efficiency.

2. A vibrating needle point-oil-mercury coherer with telephone receiver.

Mr. R. KERR, F.R.A.S. A Torsion Spring for transference of Energy (exhibited on behalf of Prof. L. R. Wilberforce, of University College, Liverpool).

A spring is rigidly fixed to an unyielding support, and, depending, carries a weight of the same diameter, which is adjusted by bolts in such a manner that when the two principal periods of vibration are as nearly equal as possible the transference of energy from an up-and-down motion to a horizontal twisting motion is complete. One pull of the spring is sufficient to cause these motions to be repeated for twenty minutes.

Messrs. WALLACH BROS. Oxygen Rescue Apparatus and other appliances.

1. The "Evertrusty" oxygen apparatus, used by the rescue parties at Courrières, consisting of two oxygen cylinders filled with oxygen, two regenerators through which the vitiated air passes and is regenerated, and which at the same time serve the purpose of ascertaining if the apparatus is in working order prior to use. The fittings and pipes include the mouthpiece, by means of which the air supply is conducted to the mouth of the operator.

2. "Evertrusty" oxygen first aid case for use in case of carbonic oxide poisoning, or after inhalation of smoke, poisonous fumes, &c., consisting of oxygen cylinder, reducing valve, pressure gauge, bag and mask, with back pressure valve.

These appliances permit of active work in atmospheres charged with gas, such as water gas, Dowson gas, Mond gas, power gas, producer gas, blast-furnace gas, after-damp, &c.

Mr. KENNETH J. TARRANT, F.R.A.S. Photographs of Electrical Discharges, at Atmospheric Pressure and in *vacuo*.

A series of photographs showing the characteristic discharge from an interrupted continuous current and a high frequency oscillating one, also between parallel conductors both insulated and bare, of varying capacity, and with various condenser conditions. Also the apparently spiral (vortex) nature of the discharge both in air and *vacuo*.

Mr. EDWIN EDSE and Mr. EDGAR SENIER. Specimens of Colour Photographs, and Photomicrographs.

1. Lippmann spectrum photographed bleached after Neuhaus's method. The colours seen by reflected light are very bright, and the complementary colours are seen by transmitted light.

2. Colour photograph produced by exposing Lippmann film successively to two continuous spectrums, the red end of one being superposed on the blue end of the other. Two short spectrums are seen on development, crossed by fine dark lines, first observed by Neuhaus.

3. Lippmann spectrum photographs viewed, at polarising angle, through Nicol's prism. Beautiful colour changes occur as the Nicol is rotated.

4. Three-colour photographs of coloured objects, including crystals under polarised light.

5. Photo-micrographs obtained through red, green, and blue colour screens. The blue colour screen improves the resolution very noticeably.

6. Photo-micrographs obtained by the aid of Zeiss apochromatic objective, and other objectives.

7. Simple form of colour sensitometer.

Mr. C. V. BOYS, F.R.S. A Gas Calorimeter.

This calorimeter is of the Hartley or Junkers type; that is, a steady stream of water is passed through the instrument, and the rise of temperature and the rate of flow are measured. The hot gaseous products from a pair of small luminous flames pass up through a short wide chimney which is only water cooled at its base. After impinging upon a bell they pass down and up again in a space in which a motor-car radiator pipe of Clarkson's or other construction is wound. The lower coils are immersed in the water-bath which prevents the chimney from burning, but which does not keep it cool enough to cause condensation within. The water leaves through an equalising chamber above the bell. This is the instrument by which London gas is officially tested.

Sir JAMES DEWAR, F.R.S. Metallic Jacketed Vacuum Vessels.

In these metallic vessels filled with liquid air the vacuum is produced by the use of cooled charcoal. The envelopes may be made of brass, copper, nickel, or tinned iron, with necks made of a bad conducting alloy. The necks can be covered with silvered glass vacuum cylinders which act as stoppers, and at the same time utilise the cold of the slowly evaporating liquid. The efficiency of the best metallic flasks is equal to that of the average silvered glass vacuum vessels now generally used in low temperature investigations. Vessels of this type may be useful in industrial cryogenic operations, and for the storage and safe transit of liquid air and oxygen.

Mr. J. E. STEAD, F.R.S. A Triple Alloy of Tin—Antimony—Arsenic, polished and etched, showing bright curved crystals embedded in a soft matrix or eutectic.

A triple alloy containing 75 per cent tin, 20 per cent antimony, and 5 per cent arsenic. After melting and slow cooling it first yields crystals, which assume the form of semi-spherical shells, and a fusible mother substance, the last to solidify, in which, when the alloy is cold, the crystals remain embedded. The crystals contain more arsenic and antimony than the matrix; they are hard and brittle, and capable of taking a high polish. They are not easily corroded by dilute acids, and not readily tarnished. The matrix, or mother substance, containing a preponderating proportion of tin, is easily dissolved out by hydrochloric acid, leaving the curved crystals in bold relief. The specimens exhibited show the fracture and macro-structure of the polished and etched alloy, treated in such a way as to reveal the structure to the best advantage.

Mr. W. ROSENHAIN. Improved Metallurgical Microscope.

Designed for the examination of metal specimens. The base and limb are of particularly rigid construction, and the tube is rigidly attached to the limb. The stage racks on the broad flange of the limb, and is provided with a fine adjustment placed in the line of the optic axis of the microscope. The internal reflectors employed for obtaining "vertical" illumination, instead of being carried on a detachable fitting, are inserted into the tube of the instrument, and are provided with adjusting movements which allow of complete control of the lighting. Various forms of reflectors are readily interchanged without disturbing the focus of the objective. Special devices for the easy attachment and adjustment of oblique and other illuminators for low-power work are provided, while a detachable

bridge can be fitted to the stage so as to adapt it for work with transmitted light. For purposes of photo-micrography a focussing motion is provided whereby the eye-piece may be moved relatively to the objective. Metallurgical specimens are shown illustrating the effect upon resolving power produced by various forms of "vertical" illuminator, and the importance of a concentric rotating stage in this connection.

The Rt. Hon. Lord BLYTHSWOOD. Photographs of certain Arc Spectra.

The spectra were produced by means of a Blythswood concave diffraction grating, the work being undertaken as a practical test of the gratings. The radius of the grating was ten feet, the first order spectrum being photographed. The total length given is about forty inches, from λ 2100 to λ 7400. There was no screening for the violet end. The red end was taken on films dyed with pinacyanol, a screen placed in front of the slit completely eliminating the second order spectrum. Salts of the elements were fed into a carbon arc on a 100 volt circuit. The photographs were taken by Mr. Walter A. Scoble, B.Sc.

Dr. O. SILBERRAD and Mr. H. A. PHILLIPS. A Series of Picrates.

The salts of picric acid are of interest as having been the probable cause of some of the most disastrous lyddite explosions on record. The specimens exhibited were in many cases prepared in the course of an exhaustive investigation recently carried out at the Research Laboratories of the Royal Arsenal. Several of the salts exhibited have never been prepared before, and the majority have never previously been obtained pure or correctly analysed. For example, nickel picrate, according to previous investigations, is capable of forming hydrated salts containing 1, 5, or 8 molecules of water. On examining the present samples it will be seen that this salt has been obtained in 3 degrees of hydration containing $9\frac{1}{2}$, 6, and 2 molecules of water respectively, the anhydrous salt being obtained on drying at 150° C.

Dr. W. MARSHALL WATTS. Binocular Spectroscope.

The Binocular Spectroscope might otherwise be described as a spectroscopic opera-glass. It consists of a field-glass, or other form of binocular, in front of the object-glasses of which two exactly similar transparent diffraction-gratings are mounted on optically worked plane-glass. As the instrument has neither slit nor collimator it is applicable, in the first instance, only to luminous objects of definite form, such as vacuum tubes. For ordinary observations of flame-spectra, or spark-spectra, a metal, or ebonite plate, with a slit, in front of the Bunsen or spark is employed. The use of both eyes in spectroscopy constitutes a new departure. The whole spectrum is seen at once, the bright lines stand out in apparent relief, and observations can be made with greater comfort and success, especially with faint spectra.

Sir WILLIAM CROOKES, F.R.S.

1. The Ultra-violet Spectra of the Metals, photographed with a quartz train of five double prisms. The spectrum of pure iron used as a standard.

Aluminium, antimony, arsenic, barium, bismuth, cadmium, caesium, calcium, carbon, cerium, chromium, cobalt, copper, europium, gadolinium, gold, indium, iron, iridium, lithium, lead, magnesium, manganese, mercury, molybdenum, nickel, nitrogen, osmium, palladium, platinum, potassium, praseodymium, radium, rhodium, rubidium, ruthenium, scandium, samarium, silicon, silver, strontium, tantalum, thallium, thorium, tin, titanium, tungsten, uranium, vanadium, ytterbium, yttrium, zinc.

Meteorites.—"Thunda," "See Lasgen," "Narraburra," "Canyon Diablo," "Boogaldi."

Ferro-alloys.—Fe 82.3, Va 13.4, Al 3.2, Si 1.1. Fe 86.6, Va 13.4. Fe 70.9, Va 29.1. Fe 10, Va 90. Fe, Ti. Fe 61.12, Wo 30.5, Cr 8.32. Fe 92.7, Mn 7.3. Fe, Mn. Fe, Si. Fe 25, Si 75.

Steel.—Chrome steel, Cr 3.5 per cent. Nickel steel,

Ni 3.06 per cent. Nickel-chrome steel, Ni 4.08, Cr 1.7 per cent. Tungsten steel, Wo 3 per cent.

2. Fifty Stereoscopic Photographs, taken by Sir W. Crookes on the occasion of the visit of the British Association to South Africa in the autumn of 1905.

Mr. G. F. HERBERT SMITH. A Refractometer for Liquids.

By means of this compact and portable form of refractometer the refractive indices of liquid and semi-liquid substances may be easily and quickly determined in sodium light to the fourth place of decimals. The attached thermometer gives the temperature at which the readings are taken, and the simple form of water-jacket permits of observations being made at temperatures differing from that of the surrounding air. The range and the degree of accuracy may be varied to suit the particular work for which the instrument is required. The principle of the optical arrangements is identical with that employed in the exhibitor's refractometer for crystals and faceted gemstones. The instrument has been constructed by Mr. J. H. Steward.

Prof. W. F. BARRETT, F.R.S. Entoptiscope, for the Self-examination of Obscurities and Defects within the Eye.

The instrument enables anyone to observe, delineate, and measure an obscurity however minute within the path of the rays in the eyeball, and affords an admirable method for the study of pupillometry of ocular parallax, and of entoptic phenomena generally.

Dr. G. T. MOODY. Specimens illustrating the indifference of Oxygen towards Iron in presence of water, and the effect of the admission of Carbonic Acid.

1. Specimen of soft Swedish iron which has been in contact with air and water both completely freed from carbonic acid for five weeks. During this period approximately 56 litres of air passed over the iron, exposing the metal to a volume of oxygen exceeding thirty times the amount required to completely convert it into ferric oxide.

2. Specimen of the same iron which had remained perfectly bright after exposure for three weeks to water and 32 litres of air, freed from carbonic acid. At the end of the period named, air cleaned by its passage through a tower packed with moist pumice-stone, but containing its normal amount of carbonic acid, was admitted. After seventy-two hours, during which time approximately 16 litres of air passed through, and when considerable rusting had occurred, the tube was sealed.

Prof. WYNDHAM DUNSTAN, F.R.S.

1. New or Rare Minerals from Ceylon.

Many of these minerals have been collected during the progress of the Mineral Survey now proceeding in Ceylon in connection with the Imperial Institute. Others have been found in river gravels sent for examination to the Imperial Institute. These minerals illustrate the wide distribution of thorium in Ceylon.

(1) *Giekielite*; (2) *Picroilmenite*.—Magnesium iron titanates. These minerals have been investigated at the Imperial Institute and described by Messrs. T. Crook and B. M. Jones in a paper recently contributed to the Mineralogical Society.

(3) *Pleonaste (Ceylonite)*.—A variety of spinel. Iron magnesium aluminate.

(4) *Zirkelite*.—Iron and calcium titanate and zirconate with thorium or uranium dioxides. Some specimens contain as much as 20 per cent of thorium.

(5) *Tscheffkinite*.—Silicates and titanates of cerium and the allied metals. It contains about 5 per cent of thorium.

(6) *Æschynite*.—Principally niobates and titanates of cerium and the allied metals containing about 5 per cent of thorium.

(7) *Thorianite*.—Principally oxides of thorium and uranium. It contains, as a rule, about 79 per cent of thorium (see *Proc. Roy. Soc.*, 1905).

(8) *Thorianite from Galle*.—This differs from the thorianite previously examined in containing more uranium dioxide, which has replaced some of the thorium. It is

therefore intermediate in composition between thorianite and uraninite. A paper dealing with the composition of this mineral has been recently communicated to the Royal Society by Prof. Dunstan and Mr. B. M. Jones.

(9) *Uraninite*.—Principally oxides of uranium containing also oxides of thorium and other metals. This specimen contains 9 per cent of thorium.

(10) *Thorite*.—Chiefly thorium silicate, containing 70 per cent of thorium.

(11) *Monazite*.—Phosphates of cerium and the allied metals. It contains about 10 per cent of thorium, and is thus richer in thorium than the monazite usually found in Brazil and North Carolina.

(12) *Allanite*.—Silicates of aluminium, iron, calcium, cerium, and the allied metals. It contains 1.5 per cent of thorium.

(13) *Fergusonite*.—Principally yttrium niobate, containing 2 per cent of thorium.

(14) *Sand from Niriella*.—Containing ilmenite, garnet, zircon, rutile, monazite, cassiterite, thorianite, and gold.

2. Minerals from Canada.

(15) *Cobalt, Nickel, and Silver Ore*.—Discovered recently at Haileybury, 227 miles north of Toronto. The minerals present are smaltite, CoAs_2 ; chloanthite, NiAs_2 ; niccolite, NiAs ; cobaltite, CoAsS ; dyscrasite, Ag_3Sb ; erythrine, $\text{CO}_3\text{As}_2\text{O}_8 \cdot 8\text{H}_2\text{O}$; annebergite, $\text{Ni}_3\text{As}_2\text{O}_8 \cdot 8\text{H}_2\text{O}$; heterogenite, $\text{CoO} \cdot 2\text{CO}_2\text{O}_3 \cdot 6\text{H}_2\text{O}$; arsenolite, As_4O_6 ; and native silver.

Dr. P. E. SHAW. An Electrical Measuring Machine.

End standards of length have hitherto been gauged by measuring machines of the Whitworth type. They act by mechanical touch. The chief objections to them are:—

1. Since mechanical touch is insensitive, it is impossible to work accurately with definite point contacts, but plane measuring ends are used. These planes are defective as to planeness and as to parallelism with one another. 2. Again, since mechanical touch is insensitive, comparatively large strains are produced in the micrometer screws and nuts at each measurement. 3. No means are provided by which the experimenter can adjust or calibrate the machine as he should do when it arrives from the maker and periodically afterwards. In the electric machine it is claimed that each of the above objections is avoided for:—1. Electric touch provides great sensitiveness, and point-to-point contact is possible. 2. No false assumptions are made as to perfection of the plane faces. 3. Strain in the machine is negligible. 4. Each part of the machine can be adjusted. The errors in gauges are measured by this machine. In some cases they are considerable.

Prof. SILVANUS P. THOMPSON, F.R.S., exhibited and described the Electric Production of Nitrates from the Atmosphere.

Various processes have been devised for the synthesis of the nitrogen and oxygen of the atmosphere by means of electric discharges. These all start from the original observations of Cavendish in the years 1781 and 1786. Recently there have been established commercial processes for the electric production of cyanamide by the process of Dr. Franck, and of calcium nitrate by the process of Birkeland and Eyde. The latter has been at work for over a year at Notodden, in Norway. The special feature is the furnace in which the synthesis is effected, the electric discharges being produced by alternating currents at about 3000 volts between copper electrodes placed between the poles of a large electromagnet. There results a roaring disc of flames about 6 feet in diameter, through which air is gently flown. The nitrous vapours with which the air is thus charged are passed through absorbing towers, where they are absorbed in water and in quicklime, the whole of the products being converted into calcic nitrate which is concentrated. The product is used for agriculture (being equal in value to Chili saltpetre) and in the dye-stuff industry. The power is furnished by waterfalls. One kilowatt may be taken as able to produce 500 kilograms of nitrate, at least, per annum.

CORRESPONDENCE

THE TEMPERATURE OF SOLUTIONS HEATED BY OPEN STEAM.

To the Editor of the Chemical News.

SIR,—Might I point out in reply to Mr. Frank Spence (CHEM. NEWS, xciii., 68) that in the *Reports of the British Association*, 1869 and 1870, in which Mr. Peter Spence's experiments are described and to which my reference applies, no theoretical explanation of the phenomenon in question is given. That this was subsequently published in the *Brit. Mer. Gazette* in 1878 was not known to me, but in any case that journal can hardly be considered a suitable medium for the publication of scientific information.

I did not state that the first person to theoretically explain the matter was Dr. Claassen. What I said was that Dr. Claassen's paper contained the only other reference thereto which I had seen.

Since my paper was published I have seen a number of other references to the subject which it may be of sufficient interest to quote. These are:—"Watts' Dict. of Chem.," 1881, vol. viii., Pt. II., Third Suppl., p. 947; "On Steam and Brines," J. Y. Buchanan, F.R.S., *Trans. Roy. Soc. Edinb.*, 1898-9, xxxix, iii., p. 529; "Physical Chemistry," James Walker, 1901, p. 84. In the above volume of "Watts' Dictionary" it is stated that "the heating of saline solutions to their boiling-points by the action of aqueous vapour at 100° was noticed more than fifty years ago by Faraday and by Gay-Lussac" (*Ann. Chim. Phys.*, 1822).

Mr. Peter Spence deserves all credit for his independent discovery, and it is surprising that so striking and interesting a phenomenon is so little known amongst chemists generally.—I am, &c.,

THOS. STEEL.

Sydney, New South Wales.
April 2nd, 1906.

NOTICES OF BOOKS.

Practical Electrochemistry. By BERTRAM BLOUNT, F.I.C., Assoc. Inst. C.E. Second Edition. London: Archibald Constable and Co., Ltd. New York: The Macmillan Company. 1906.

THE second edition of this work has been entirely brought up to date, and enlarged to a considerable extent, the descriptions of many new processes and of modifications of old methods having been added. For instance, we may mention the new section on the electro-metallurgical production and treatment of iron and steel, in which region during the last five years the earlier experimental difficulties have been overcome, so that the industry bids fair to become one of great importance. Thus the book now gives a thoroughly complete and systematic survey, from the most modern point of view, of the principles and practice of all the most important electrochemical processes employed industrially, and, as in the former edition, the practical aspect of the advantages to be gained by the use of electrochemical methods is emphasised.

Physical Chemistry for Electrical Engineers. By J. LIVINGSTON R. MORGAN, Ph.D. First Edition. New York: John Wiley and Sons. London: Chapman and Hall, Ltd. 1906.

THIS book is intended especially for those who wish to acquire a slight knowledge of physical chemistry in as short a time as possible, and as far as it goes it appears to fulfil its purpose. The difficulty of writing such a

sketch of a wide subject has been surmounted on the whole successfully by the author, whose work shows plain traces of his indebtedness for many of his ideas and explanations to Prof. Ostwald's "Vorlesungen über Naturphilosophie." The book will perhaps serve as an introduction to the study of larger and more complete works on the same subject, although it is hardly interesting enough to awake much enthusiasm in a beginner. Certain important branches, e.g. solutions, are treated comparatively fully, and the collection of problems will be found useful in consolidating the student's knowledge of the general principles of physical chemistry. The addition of an index would add to the value of the book.

The Origin of Life. By JOHN BUTLER BURKE. London: Chapman and Hall, Ltd. 1906.

GREAT interest was aroused, especially among the general public, when the results of Mr. J. B. Burke's experiments were first announced some months ago, and it seems likely that this book which he has just published, giving further information on the same subject, will be purchased and read by a wider and less highly trained circle of readers than that which generally concerns itself with scientific works. It is to be feared that the uninitiated reader will close the book without having acquired a very high opinion of it as a contribution to our knowledge of the physical basis of life. He will have read a large amount of theoretical discussion, especially in regions in which speculation is safe and contradiction impossible, but he will have learned very little more precise information about those mysterious radiobes than was to be gathered from the first articles in which they were described. The test of the vitality of the radiobe is, in the author's opinion, the stoppage of growth and the beginning of the disintegration process; and since special emphasis is laid upon this latter phenomenon, we might expect to find details and definite measurements, which, however, we look for in vain. While the author displays a powerful imagination, without which the scientific investigator cannot hope to proceed very far, he appears to be unable to present theory and fact to his readers so that a sense of the true proportion between them is maintained, and when he elaborates his theory, claiming that some of the processes of nature have been imitated by the artificial production of cells, and that he has found the bridge between the organic and inorganic worlds, we feel that he is basing an unwieldy and top-heavy structure upon a shallow and inadequate foundation.

Petrogenesis ("Petrogenesis"). By Dr. C. DOELTER. Braunschweig: Friedrich Vieweg und Sohn. 1906.

THIS book is the thirteenth of the series of monographs on scientific and mathematical subjects, "Die Wissenschaft." The author has a very wide knowledge of petrography and mineralogy, and has prepared an exhaustive treatise on the origin of rocks, dealing first and most fully with eruptive formations, and then passing to crystalline schists and sedimentary deposits. The great advances which have recently been made in our knowledge of the origin of rocks are to be ascribed to the spread of the use of the methods of physical chemistry, and these are treated at length, the experimental parts of the subject being made especially prominent. The question of the nature of rocks can be satisfactorily explained only when the mineral constituents and the chemical composition have been thoroughly investigated in conjunction with the geological occurrence, and these subjects the author is thoroughly competent to deal with in the most enlightened spirit. A pleasing feature of the book is the full recognition of the work of the geologists and chemists of all nations, periodical literature in many languages having been carefully studied and abstracted, so that a most comprehensive survey of the whole subject is given.

Microscopes and Microscopical Accessories. Jena: Carl Zeiss. 1906.

THE thirty-third catalogue issued by the firm of Carl Zeiss contains details of the objectives and oculars, stands, and accessory apparatus, manufactured by this famous firm. Many new forms of stands are advertised, among them a simpler kind for laboratory use, to which we may call special attention, and large reductions are notified in the prices of achromatic objectives, Huygenian oculars, &c., while in the case of ruled gratings and heating chambers, slight advances have been made in the prices. Magnifiers and dissecting stands are not included in this list, a special catalogue of them having been issued. All those who are contemplating buying a microscope or accessories should make a point of consulting this list, which is fully illustrated, and contains specifications of complete outfits as well as information relating to all kinds of separate pieces of microscopical apparatus. We understand that Messrs. Zeiss will forward a Catalogue to any of our readers who apply for the same.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances de l'Académie des Sciences. Vol. cxlii., No. 16, April 17, 1906.

Pure Ferro-molybdenums.—Em. Vigouroux.—In a recent paper the author describes two general methods of preparing pure ferro-molybdenums. He now investigates their composition and properties. The residue, Fe_3Mo_2 , which remains when two of the alloys are exhausted in the cold, either by hydrochloric acid or copper chloride in hydrochloric solution, has a density of 9.16, and is non-magnetic. The body FeMo , which can be separated from alloys containing between 54 and 63 per cent of Mo, has a density 9.01, and is also non-magnetic. It becomes incandescent in chlorine at 285° , and in oxygen at a red heat. FeMo_2 can be isolated from alloys containing between 64 and 67 per cent Mo, and has density 9.41.

Characteristic Reaction of Ethyl Glyoxylate. Action of Ammonia on this Ether and its Derivatives.—L. J. Simon and G. Chavanne.—Ethyl glyoxylate, under the action of pure aqueous ammonia, gives a precipitate which is white at first, and, after passing through various tints, finally becomes bluish black. At the same time the ammoniacal liquor becomes dark red. This succession of colorations takes place rapidly when the liquid is hot and slowly when cold. When the ethyl glyoxylate is present in very small quantity the liquid remains limpid, but even traces of ether bring out the colour. Ammonia can be replaced by its carbonate, CO_2 being evolved during the reaction. The coloration is due to a black substance which the authors isolate and analyse: $2\text{COH} \cdot \text{CO}_2\text{C}_2\text{H}_5 + 3\text{NH}_3 = 2\text{C}_2\text{H}_5\text{OH} + \text{C}_4\text{H}_9\text{N}_3\text{O}_4$. It is found to be a true ammoniacal salt.

Acid Properties of Amidon.—E. Demoussy.—The author's experiments show amidon to have all the characteristics of a weak acid. It may be compared with carbonic acid, in that it resembles the other hydrates of carbon. Similarly, it contracts with the metallic hydrates of compounds dissociable with water, and will absorb small quantities of neutral salts. These properties probably assist in the absorption of mineral matters by plants, and particularly contribute to the mineralisation of organisms containing amyl reserves.

No. 17, April 23, 1906.

The Diffusion of Solutions and their Molecular Weights.—Michel Yégonow.—In order to determine the velocity of movement of oxygen and hydrogen sulphide in nutritive media and also in water for micro-biological researches, and finding no direct method, the author was obliged to attack the problem indirectly. He first supposed that definite relations can exist between the coefficient of diffusion (K) and the velocity of movement (v) in a given section for solutions having the same osmotic pressure; that is to say, equimolecular solutions. He makes experiments on pure absolutely transparent 10 per cent gelatin solution, and finds (1) that the proportion between the coefficient of diffusion (K) and the velocity of motion (v) is a constant for equimolecular solutions of all substances; (2) that when the concentration (x) varies in geometric progression the velocity v varies in arithmetic progression.

Atomic Weight and Spark Spectrum of Terbium.—G. Urbain.—The author has previously explained his method of isolating terbium in a state of purity, and has also shown that he was unable to assign a characteristic spectrum to this body. In order to determine the atomic weight of terbium he estimates the water in the octohydrated sulphate $(\text{SO}_4)_2\text{Tb}_3 \cdot 8\text{H}_2\text{O}$. His numbers are very concordant, and the mean of five experiments is 159.22 if $\text{O} = 16$. A further examination of the spark spectrum shows it to be very rich in lines. The most characteristic are given in a table.

Estimation of Cadmium in Volatile or Organic Salts.—H. Baubigny.

NOTES AND QUERIES.

* Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Reviving Faded Ink.—I should be greatly obliged for particulars of any chemical which will revive (if only temporarily) red and black ink. I have a very old document (parchment), and the writing, which is in both red and black ink, has in many places quite faded away.—S. G. W. MANN.

MEETINGS FOR THE WEEK.

- MONDAY, 21st.**—Society of Arts, 8. (Cantor Lectures). "Heraldry in relation to the Applied Arts," by George W. Ebe.
— Society of Chemical Industry, 8. "The Problem of the Electro-chemical Fixation of Nitrogen," by P. A. Guye.
- TUESDAY, 22nd.**—Royal Institution, 5. "Glands and their Products," by Prof. William Stirling, M.D., &c.
- WEDNESDAY, 23rd.**—Society of Arts, 8. "The General Supply of Electricity for Power and other Purposes," by James N. Shoolbred, B.A.
- THURSDAY, 24th.**—Royal Institution, 5. "Man and the Glacial Period," by Prof. W. J. Sollas, F.R.S.
— Society of Arts, 4.30. "The Persia of Persia," by Major Percy Molesworth Sykes.
- FRIDAY, 25th.**—Royal Institution, 9. "Compressed Air and its Physiological Effects," by Leonard Hill, M.B., F.R.S.
— Physical, 5. "Colour Phenomena in Photometry," by J. S. Dow. Exhibition of an Automatic Arc Lamp, by H. Tomlinson and Rev. G. T. Johnston. "Theory of Moving-coil and other kinds of Ballistic Galvanometers," by H. A. Wilson. Exhibition of Bifilar Galvanometer free from Zero Creep, by A. Campbell.
- SATURDAY, 26th.**—Royal Institution, 3. "The Old and the New Chemistry," by Prof. Sir James Dewar, D.Sc., F.R.S., &c.

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2 m/m.	100° C.	1520° C.
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40	500	1460
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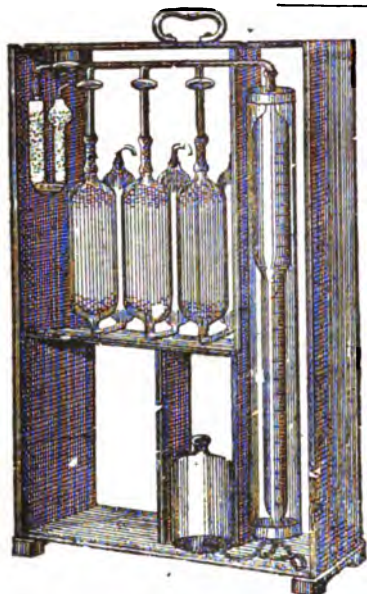
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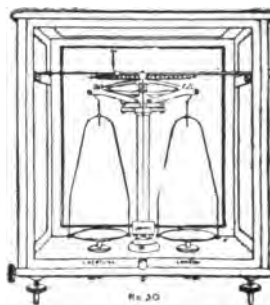
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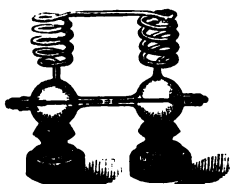
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THE CHEMICAL NEWS.

VOL. XCIII., No. 2426.

ON THE DISTRIBUTION OF RADIUM IN THE EARTH'S CRUST, AND ON THE EARTH'S INTERNAL HEAT.*

By the Hon. R. J. STRUTT, F.R.S., Fellow of Trinity College, Cambridge.

I. Introduction.

PROFESSOR RUTHERFORD has given a calculation which suggests that there may be enough radium in the earth to account for the temperature gradient observed near the surface ("Radio-activity," p. 494, 2nd Edition).

The question is of great interest from a cosmical point of view. For if we find that the earth's internal heat is due to radio-activity, and if we assume, as has been usual, that this heat is due to some vestiges of the cause operative in the sun and stars, it would follow that these latter are heated by radio-active changes also.

Prof. Rutherford's calculation was based on some data given by Elster and Geitel on the amount of radium emanation which diffused out from a sample of clay. These data were obtained at a time when the quantitative determination of minute amounts of radium was not well understood, and are, moreover, inadequate to give any general idea of the average amount of radium in the earth's crust. I have therefore made an extensive investigation of the amount of radium in various representative rocks. This forms the subject of the present paper. The results are very surprising. Considerable detail will therefore be given in order to enable readers to judge whether there is any probability of these results being substantially incorrect.

2. Choice of Material.

The earth's crust, which is alone accessible to us, consists of igneous rocks, and of sedimentary material which results from the action of geological changes upon these rocks. No doubt the average radium content of the original igneous rocks might be inferred fairly well from the examination of a large number of sedimentary ones. It is, however, much more satisfactory to examine the igneous materials directly, for then they can be classed among themselves as to their radium content.

I have examined a few sedimentary rocks, but do not attach much importance to them, and rely chiefly on results obtained with original igneous material for determining the average radium content of the earth's crust. The results with regard to sedimentary rocks will be given in a future paper. I hope also to determine which of the numerous minerals contained in igneous rocks carry the radium, a subject not touched in the present communication.

Meteorites have a special interest of their own. A few determinations have been made on them, and are incidentally included in this paper.

3. Method of Determining Radium Content.

Radium in the rocks was quantitatively determined by means of its emanation. A solution of the rock was stored till the emanation had accumulated. This latter was then extracted by boiling, and introduced into an electroscope. The increased rate of leak produced was a measure of the amount of radium present. This measure was made absolute by going through the same process with a uranium mineral of known radium content.

In order to make certain of extracting the emanation quantitatively, it is essential to decompose the rock completely by chemical agency. In the case of limestones and

metallic meteorites, this can be effected by solution in hydrochloric acid, but in the case of siliceous materials, fusion with alkaline carbonate is necessary.

The standard procedure was as follows:—Fifty grms. of the rock was broken up in an iron mortar, and then finely ground in an agate one, until it would pass through a sieve of 60 threads to the inch. This process could be carried out by an unskilled assistant in less than an hour, and ensure easy decomposition of the rock.

Two hundred and fifty grms. of a mixture of anhydrous sodium and potassium carbonates was melted in a large platinum basin. For this purpose the basin was surrounded with an extemporised furnace casting of asbestos millboard, and heated from below by means of a gas blowpipe. The blowpipe was supplied with air from an automatic blowing apparatus worked by water pressure. As soon as the carbonate was melted, the rock powder was thrown on to its surface in small portions at a time until it had all been added. The fusion was usually continued for about an hour after all effervescence was over.

The residue was then digested with hot water to dissolve out the alkaline silicates and carbonates. The portion insoluble in water was dissolved in hydrochloric acid. Some silica always separated from the acid solution, and was allowed to remain floating in it.

The two solutions, aqueous and acid, were set aside in separate flasks closed with indiarubber stoppers. Mixing them was avoided, because of the bulky and unmanageable precipitate of silica which would have been thrown down.

Decomposition with hydrofluoric acid has some advantages over the use of sodium carbonate. It requires, however, more minute pulverisation of the material, and is more costly in practice owing to difficulties of carriage and storage. After one or two trials its use was abandoned.

The two flasks containing the respective solutions were allowed to stand for some determinate number of days. A week was the minimum. More commonly a fortnight or three weeks was allowed. During this time, any radium contained in the rock was generating emanation. After three weeks the quantity has practically reached a maximum. The fraction of this maximum generated in any lesser period could be calculated from the equation for the rise of activity, originally given by Rutherford and Soddy.

I described a method for quantitatively extracting the emanation in *Proc. Roy. Soc.*, 1905, A, vol. lxxvi., p. 89. An improved modification of that method has been employed in the present investigation. The flask A (see figure) contains the solution. The accumulated emanation is at first partly dissolved, partly contained in the air which occupies the upper part of the flask. The flask is uncorked and attached to the lower end of the condenser B as rapidly as possible, so as to avoid loss of emanation. A is then boiled to expel radium emanation. The steam which issues is condensed in B, and drops back. The air charged with emanation passes out into the gas-holder C, displacing the water which previously filled it. This boiling is continued for one hour. At the end of that time, the cooling water is run off from the jacket of B. Steam is allowed to pass so as to wash out all air and emanation from A, and from the connecting tubes into C. The indiarubber connection at D is then nipped,* and the burner under the flask immediately withdrawn.

In this way the emanation is collected in the gas-holder C. It merely remains to transfer it to the electroscope when cold. The latter is exhausted, and the emanation allowed to pass into it through the stopcock E and a drying tube. Air is then admitted to the electroscope up to atmospheric pressure. After an interval of three hours, to allow the active deposit to form, the rate of leak is read.

The normal leak of the electroscope was repeatedly determined in the course of the investigation.

The following are the values in scale-divisions per hour:—24, 25.3, 24.1, 24, 23, 20.6, 21.7, 22.3, 22.5, 22.1;

* A Paper read before the Royal Society, April 5, 1906.

* To do this conveniently, it is very desirable to have the pinchcock attached to a firm support, so that it can be screwed up with one hand.

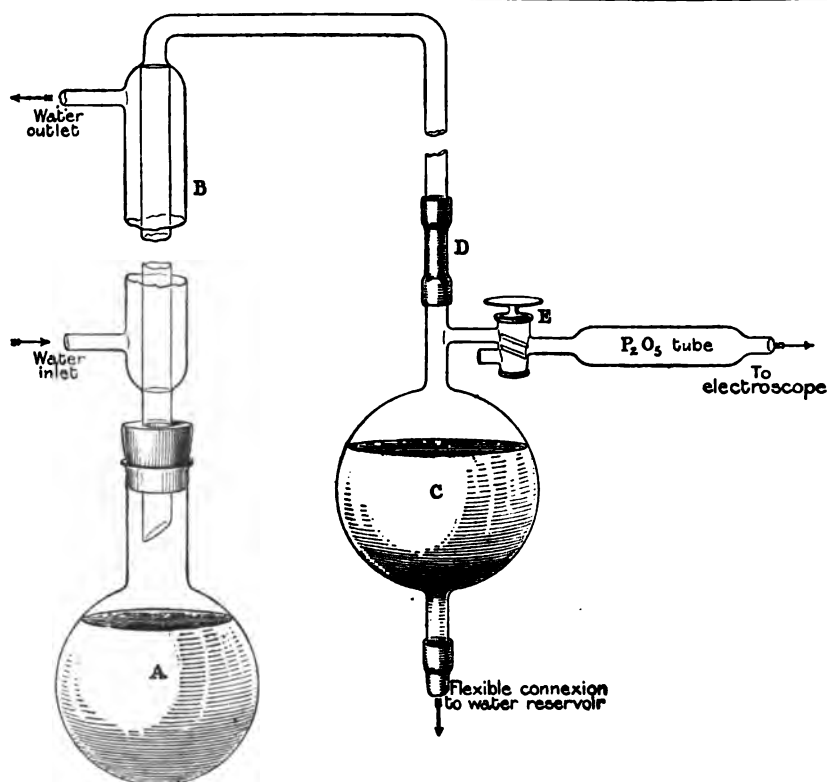


FIG. 1.

TABLE I.—Standardisation Experiments.

Mineral.	Percentage of uranium (metal).	Quantity (gram.).	Rate of leak* due to emanation accumulated in one day.	Constant deduced.
Torbernite	51.0	0.0076	214.0	2.3 × 10 ⁻¹²
Torbernite, same sample	51.0	0.0076	209.0	2.36 × "
Torbernite	51.0	0.0046	116.5	2.56 × "
Torbernite, same sample	51.0	0.0046	113.0	2.64 × "
Pitchblende	62.4	0.0063	207.0	2.41 × "
Pitchblende, same sample	62.4	0.0063	202.0	2.45 × "

* Corrected for normal leak of the electroscope.

mean, 23 nearly. These values were taken soon after exhaustion of the electroscope and admission of air.

If the instrument was left closed, the leak was found to have risen appreciably after the lapse of a day. A similar effect has been noticed at the Cavendish Laboratory, and is, I believe, under investigation there. It does not come into question here, since time was never allowed for it to enter.

In work of this kind, when the effect to be looked for is small, it is most necessary to make certain that the emanation really comes from the material under investigation, and not from any extraneous source. I was deceived in this way in concluding that mercury gave off an emanation. In the present case every precaution was taken.

The laboratory had never had radio-active materials introduced into it. Solutions of the reagents employed were separately tested for emanation, after they had stood closed for a fortnight, with the following results:—

	Rate of leak.
Sodium carbonate, 250 grms.	23.9
Potassium carbonate, 250 grms.	24.2
Hydrochloric acid, 500 c.c.	24.0
Water (Cambridge supply), 2000 c.c.	24.0

In none of these cases does the leak measurably exceed that normal to the electroscope. It may therefore be concluded that the reagents used to decompose the rocks are not responsible for the emanation obtained.

Cambridge tap-water was used to make the solutions and to fill the gas-holder c (figure). This water originally contains dissolved emanation, as Professor J. J. Thomson has shown. It was therefore carefully boiled to expel this before use. The test given above was made on water which had already been boiled before setting it aside, and shows that no measurable quantity of emanation is generated by dissolved material when the original supply has been expelled. The (boiled) water in the gas-holder was changed after each experiment.

Rutherford and Boltwood have determined the radium content of uranium minerals in absolute measure (*Am. Journ. Sci.*, 1905, p. 55). They find that the radium associated with 1 grm. of uranium is 7.4×10^{-7} grm. I use this value rather than my own, because they tested the heat production of their standard radium, while I had no test for the purity of mine (*Proc. Roy. Soc.*, 1905, A, lxxvi., p. 88).

The uranium minerals used for standardisation were

torbernite (copper-uranium phosphate), containing 60 per cent U_3O_8 (= 51 per cent uranium), and pitchblende, containing 73.5 per cent U_3O_8 (= 62.4 per cent uranium).

The rate of leak due to the emanation produced by a few m.grms. of each of these in *one day* was determined. From this the quantity of radium was deduced, which would in an *indefinite time* produce enough emanation to give a leak of one division per hour. This is the constant given in the last column of Table I.

The mean value for the constant is 2.45×10^{-12} ; or, in other words, a leak equal to one scale-division per hour represents 2.45×10^{-12} gm. of radium in the sample examined, if the latter has had time to produce its maximum amount of emanation.

Two specimen determinations will now be given in detail, the first of granite from the Cape of Good Hope, the second of olivine rock from the Isle of Rum. These are respectively representative of high and low radium content.

Cape Granite (50 grms.).—Solutions stood from March 21 to March 26, giving 60 per cent of the maximum amount of emanation.

	Scale-div. per hour.	Corrected.
Emanation from acid solution ..	103	80.0
Emanation from alkaline solution ..	31	8.0
Total	88.0 scale-divisions per hour.	

Thus the equilibrium amount of emanation would give 146 scale-divisions per hour, or per gm. of rock $\frac{146}{50} = 2.92$ scale divisions per hour. Thus 1 gm. of rock contains $2.92 \times 2.45 \times 10^{-12}$ gm., or 7.15×10^{-12} gm. radium.

Olivine Rock, Isle of Rum (50 grms.).—Solutions stood from January 31 to February 19, giving practically the equilibrium amount of emanation.

	Scale-div. per hour.	Corrected.
Emanation from acid solution ..	34.0	11.0
Emanation from alkaline solution ..	25.8	2.8
Total	13.8 scale-divisions per hour.	

One gm. of rock would give 0.276 scale-divisions per hour, and contains $2.45 \times 0.276 \times 10^{-12} = 0.676 \times 10^{-12}$ gm. radium.

This last example represents, as mentioned above, nearly the lowest radium content encountered among igneous rocks. It will be noticed that the leak produced by the emanation is, even in this unfavourable case, about half that normal to the electroscope, and is, therefore, quite well marked. It will be noticed also that the alkaline solution contains only a small proportion of the total radium present in the original rock.

(To be continued).

A REVISION OF THE ATOMIC WEIGHTS OF SODIUM AND CHLORINE.*

By THEODORE WILLIAM RICHARDS
and
ROGER CLARK WELLS.

(Continued from p. 230).

THE RATIO OF SODIC TO ARGENTIC CHLORIDE (continued).

In most experiments the residue of asbestos shreds was found, after ignition, to contain a trace of silver. This was undoubtedly produced from the decomposition of silver chloride during ignition, the latter salt having passed through the Gooch as exceedingly fine particles or having precipitated, in time, upon the asbestos shreds. The amount of this silver, as well as the amount of asbestos shreds, varied according to the efficiency of the Gooch mat and the fineness of the holes of the Gooch filter—the smaller those holes the better. The loss of chlorine upon ignition was determined in sixteen experiments, chiefly in those upon the

synthesis of silver chloride. This was done by determining the trace of reduced silver by Volhard's process, using 1/100 normal solutions. The average of determinations, which varied from 0.00 to 0.07 m.grm., amounted to only 0.02 m.grm.; this correction was applied. Although negligible, its magnitude had to be determined in order that one might be assured of safety in neglecting it.

With the asbestos at our disposal, the prepared Gooch crucible was not at all hygroscopic. An hour's standing in the balance case had no effect upon its weight. Even upon ignition, the mat, weighing less than 0.03 gm., lost only a negligible amount—and it was never heated above 150° in the actual analyses.

As has already been said, no chloride could be found by the nephelometer in any of the wash waters containing any considerable amount of argentic nitrate. Hence, after filtration through the small filters, these were cast aside and the small solubility correction of 0.03 m.grm. per litre applied to them.

The next two or three wash waters, containing only small amounts of nitrate, were collected separately, because they contained traces of chloride also. The subsequent wash waters—free from nitrate, but containing much chloride—were all united. The chloride in both separate portions was carefully determined by means of the nephelometer—that is, by comparison of opalescence. The two portions, one containing traces of argentic nitrate and one free from salt, must be estimated separately, because if all the wash waters are mixed together the opalescence usually occurs prematurely—a circumstance which interferes with the accuracy of the nephelometric comparison.

The amount of silver chloride thus found in the wash waters was added to the weight of that collected on the Gooch crucible.

Any accidentally included foreign substance—for example, silica from the glass-stoppered flasks, dust from the air, or occluded sodic nitrate—which would make the obtained weight of silver chloride too great, would make the computed atomic weight of sodium too low. Since our results were lower than Stas's, particular attention was paid to these and similar possibilities. One question especially seemed to demand an answer, namely, that concerning the possibility of the abrasion of powdered glass from the ground-glass stopper of the precipitation flask.

A newly-ground stopper sheds glass noticeably. It must always be polished and rubbed a long time until the loose particles are thoroughly dislodged. A flask of soft glass never ceases to grind away slightly every time the stopper is inserted. The large 2-litre Erlenmeyer flasks used in the final experiments were of very hard insoluble glass, with rather wide necks, very suitable for precipitation and for washing. In order to test the extent of their abrasion, one of the stoppers was removed and replaced a hundred times. The resulting powdered glass was collected on a small fine filter and found to weigh 0.2 m.grm. Since, during a single determination, the stopper was not replaced over a dozen times, it is safe to infer that the error from this cause could not have exceeded 0.02 m.grm.—a negligible quantity.

Other sources of contamination were so carefully guarded against that they can hardly have been present.

The foregoing general description may be profitably illustrated by recounting the details of a single experiment before the results of all the experiments are given.

Experiment 63 (April 5, 1904; Salt J).

	Grms.
Corrected excess weight of crucible over counterpoise	0.19994
Corrected excess crucible plus fused salt over counterpoise	5.76754
Weight salt in air ($24^\circ 764$ m.m.)	5.56760
Vacuum correction	+0.00231
Weight of sodic chloride in vacuum	5.56991

* Published by the Carnegie Institution of Washington, April, 1905.

As a suitable excess of silver nitrate, 16.40 grms. were taken for precipitation. The concentration of the silver salt being about fifth normal and that of the sodic salt not much greater, the total mother-liquor was 1.4 litres. Five washings of the precipitate were made after it had stood over night, with 0.2-litre portions of water containing about 0.05 grm. of silver nitrate per litre. Washings Nos. 6 and 7 were made with pure water and collected separately. Washings 8 to 12 were made with slightly acidified water and collected separately. The whole precipitate was finally washed upon the Gooch crucible. After drying it appeared perfectly white.

Corrected excess weight Gooch crucible (dried at 150°) over counterpoise	Grms.
Corrected excess weight crucible and AgCl (dried at 150°) over counterpoise	0.21709
Weight of silver chloride in air	13.65673
Vacuum correction	+0.00100

Weight of unfused AgCl in vacuum .. 13.65773

The mother-liquors and wash water were passed through a small filter, which was ignited in a small porcelain crucible.

Corrected excess porcelain crucible over counterpoise	Grms.
Corrected excess, plus residue and ash	0.42525
Residue and ash	0.00010
Ash	0.00001
Residue, asbestos shreds	0.00009
Loss of chlorine during ignition (see p. 230) ..	+0.00001

Total residue 0.00010

A large portion of the silver chloride, 10.50 grms., was fused in a small approximately counterpoised crucible weighing about 8 grms.

Corrected excess crucible, plus AgCl, over counterpoise (a)	Grms.
After fusion, corrected excess over counterpoise	10.64846
Loss of 10.50 grms. AgCl on fusion ..	0.00077

Hence loss of total AgCl = $\frac{13.66}{10.50} \times 0.00077 = 0.00100$.

(a) In this case the counterpoise contained no argentic chloride, but in the most careful experiments the counterpoise contained as much of this salt as the test crucible.

The nephelometer revealed no chloride in the mother-liquor and first five washings; the correction of +0.07 m.grm. was applied for this solubility even in the presence of the excess of silver nitrate.

Washings Nos. 6 and 7, volume 0.220 litre in all, were found to have a trace of chloride, and its amount was estimated by the nephelometer, making comparison with two different standard tubes.

The factor for converting 1 millilitre of standard solution in a nephelometer tube into silver chloride per litre was 0.047. (See Table, next column).

For 220 cubic centimetres the weight of dissolved AgCl is $\frac{220}{1000} \times 0.00028 = 0.00006$ grm.

Similarly for washings Nos. 8 to 12 (0.94 litre) was found on the average 0.00146 grm. of argentic chloride per litre (the individual determination being 1.47, 1.41, 1.54, and 1.44 m.grms.). For 940 cubic centimetres we have $\frac{940}{1000} \times 0.00146 = 0.00137$ grm., and hence the total

Standard tube containing NaCl solution (c.c.).	Time of forming opalescence.	Heights of opalescences appearing equal.		Ratio of heights.	Soluble silver chloride.
		Standard.	Wash waters.		
0.02	15 mins.	30	100	0.30	$0.30 \times 0.02 \times 0.047 = 0.00028$ grm. per litre.
0.005	7 mins.	100	80	—	$1.18 \times 0.005 \times 0.047 = 0.00028$ grm. per litre.
	9 hrs.	100	85	1.18	

dissolved silver chloride = $0.00007 + 0.00006 + 0.00137 = 0.00150$.

Altogether we now have for the total weight of argentic chloride:—

Weight of argentic chloride (vacuum) in crucible	Grms.
Asbestos, &c., washed through Gooch crucible	13.65773
Dissolved	+0.00010
Loss of fusion	+0.00150
Corrected weight of argentic chloride ..	13.65833

Ratio,—

AgCl : NaCl = 13.65833 : 5.56991 = 100.000 : 40.7803.

Molecular weight NaCl (if Ag = 107.93 and Cl = 35.455) = 40.7803×143.385 58.473
Assumed atomic weight Cl 35.455

Hence atomic weight Na 23.018

Most of the following determinations were essentially similar to this, but a few small differences should be noted. In Experiment 27 the eight washings were extended over five days. In Experiment 64, which must be considered as the best single experiment of all, the concentrations were lowered to tenth normal, and twelve washings were made, the first within three hours, the last after a day's lapse. Considerable nitric acid was added to the first seven wash waters. The slightly higher result of this experiment probably indicates diminished occlusion of sodic nitrate.

Three experiments were rejected, two because blackening of the argentic chloride showed that the argentic nitrate in the first wash waters had not been eliminated by the subsequent washing with pure water, and one because of known loss of argentic chloride. These oppositely erring results would have no effect on the final average if they had been included; but in such a table as this it seems advisable to include only results not vitiated by known sources of error. In the following table are given the final data and results of this comparison of argentic and sodic chlorides.

The Ratio of Sodic and Argentic Chlorides.

Final Series.

Expt.	Prep.	Weight NaCl (in vacuo). Grms.	Weight AgCl (in vacuo). Grms.	Parts NaCl equal to 100.000 parts AgCl.
13.	K	3.27527	8.03143	40.781
19.	B	5.56875	13.65609	40.779
20.	A	4.18052	10.25176	40.779
22.	C	4.54319	11.14095	40.779
23.	D	1.97447	4.84196	40.778
27.	E	3.97442	9.74547	40.782
61.	H	6.69495	16.41725	40.780
62.	I	2.88692	7.07955	40.778
63.	J	5.56991	13.65833	40.780
64.	J	5.85900	14.36693	40.781
		44.52740	109.18972	Aver. 40.780

If the atomic weight of silver is taken as 107.930, and that of chlorine as 35.455, the atomic weight of sodium, calculated from the above average, will be 23.017—a result 0.14 per cent lower than Stas's.

In spite of every effort to detect some error tending to diminish the atomic weight of sodium, and thus to cast doubt on this low value, and in spite of increasing precautions and increasing skill in the details of the experimentation, the last results are no higher than the very first one. Hence further prosecution of this method seemed to be of no avail. The reason for the difference between this value 23.017 for the atomic weight of sodium and Stas's 23.048 is best discussed after the results of the comparison of sodic chloride with metallic silver have been detailed.

(To be continued).

A SYSTEM OF QUALITATIVE ANALYSIS INCLUDING NEARLY ALL THE METALLIC ELEMENTS.*

PART II.—ANALYSIS OF THE TUNGSTEN GROUP.

By ARTHUR A. NOYES.

(Continued from p. 227).

Confirmatory Experiments and References.

G. D., § 4.—See C. E., P. 35, N. 4, in regard to the separation of tungsten from titanium by digestion with $(\text{NH}_4)_2\text{S}$, and C. E., P. 38, N. 5, in regard to that by fusion with Na_2CO_3 and S.

For the separation of tungsten and titanium by fusion with KNO_3 and K_2CO_3 , see Defacqz, *Comptes Rendus*, 1896, cxxiii., 823.

A mixture of 10 m.grms. W and 300 m.grms. Ti in HF solution was treated by P. 33, 35, 36, and 38, except that the fusion with Na_2CO_3 and S was replaced by the following process:—The NH_4OH precipitate obtained after the treatment with H_2O_2 , and the evaporation with H_2SO_4 was ignited in a platinum crucible and mixed with eight times its weight of eight parts KNO_3 and two parts K_2CO_3 , the mixture kept at a dull red heat for thirty minutes; the mass was treated with water, the water evaporated off, the residue again treated with water, and the liquid filtered. The residue was dissolved in H_2O_2 and HCl, the solution evaporated with H_2SO_4 , and, after dilution, poured through a Zn column into HgCl_2 solution; no precipitate resulted. The filtrate was strongly acidified with HNO_3 and evaporated to dryness; the mass was treated with water, the solution filtered, and the (white) residue treated with NH_4OH ; only a small portion of it dissolved, but the solution, on acidifying with HNO_3 , gave a small precipitate of H_2WO_4 (1 to 2 m.grms.); the white residue gave no colour when heated with H_2O_2 and HCl.

The experiment was repeated with a mixture of 300 m.grms. W and 300 m.grms. Ti; a large grey precipitate was produced in the HgCl_2 solution after the NH_4OH precipitate had been fused with KNO_3 and K_2CO_3 .

G. D., § 5.—100 m.grms. Ti in one experiment, 5 m.grms. Nb in a second, and a mixture of 100 m.grms. Ti and 5 m.grms. Nb in a third, as oxides, were dissolved in HF, the solution evaporated three times with HNO_3 , the residue heated thirty minutes at 120° , and then warmed with 10 c.c. 3 per cent H_2O_2 and 1 c.c. HNO_3 (1.20); the TiO_2 and also the mixture of TiO_2 and Nb_2O_5 dissolved completely, but the pure Nb_2O_5 did not seem to dissolve at all.

500 m.grms. Ti (as H_2TiO_3) were dissolved in HF, the solution evaporated just to dryness with HNO_3 , the residue moistened with HNO_3 , and the latter evaporated off; 10 c.c. 3 per cent H_2O_2 and 2 c.c. HCl (1.12) were added, and the mixture heated to the decomposition-

temperature of the H_2O_2 ; in three minutes everything had dissolved. The same experiment was repeated with 2 m.grms. Nb as Nb_2O_5 , and in a separate experiment with 2 m.grms. Ta as Ta_2O_5 ; most, if not all, of the substance remained undissolved in each case, even on five to ten minutes' heating. The experiment was again repeated with a mixture of 12 m.grms. Nb and 100 m.grms. Ti; everything dissolved on three minutes' heating except a few hard flakes, and these went into solution on heating for five minutes longer. It was also repeated with a mixture of 5 m.grms. Ta and 100 m.grms. Ti; the whole residue went into solution in five minutes. It was repeated with 500 m.grms. Ti, except that no HCl was at first added with the H_2O_2 ; the residue dissolved much more slowly, and some still remained after ten minutes, but this dissolved at once on adding 2 c.c. HCl (1.12).

For the separation of titanium and uranium from iron by alkaline H_2O_2 , see P. H. Walker (*Journ. Am. Chem. Soc.*, 1898, xx., p. 515).

To a solution in 5 c.c. H_2SO_4 (1.20) of 100 m.grms. Ti in one experiment, and of 100 m.grms. Ti and 6 m.grms. Ta in another, 50 c.c. 3 per cent H_2O_2 solution were added, and this mixture was dropped rapidly from a separating funnel into a cold mixture of 50 c.c. 3 per cent H_2O_2 and 15 c.c. NH_4OH (0.90); no precipitate formed within fifteen minutes in either case, but a large yellowish white precipitate came out on standing over night at a temperature of about 10° .

G. D., § 7.—100 m.grms. Ti as TiO_4H_4 were dissolved in 10 c.c. 3 per cent H_2O_2 and 2 c.c. HCl (1.12), SO_2 gas passed in to saturation, NH_4OH added till alkaline, the solution allowed to stand three minutes, and then to the mixture a fourth its volume of HCl (1.20) was added; the whole precipitate dissolved. The experiment was repeated, substituting one m.grm. Nb for the 100 m.grms. Ti; the NH_4OH precipitate remained in large part undissolved. The experiment was repeated with a mixture of 50 m.grms. Ti and 2 m.grms. Nb, and with one of 100 m.grms. Ti and 5 m.grms. Nb. In the first case the whole of the NH_4OH precipitate dissolved; and in the second case only a slight one remained, which, when dissolved in H_2SO_4 and treated with Zn and HCl gave no colour, showing the absence of niobium.

G. D., § 8.—See C. E., P. 38, N. 4 in regard to the reduction by Zn and the HgCl_2 test in the cases of titanium and niobium.

G. D., § 9.—To two portions of 7 c.c. H_2SO_4 (1.20), NH_4OH was added till alkaline, and then 7 c.c. $(\text{NH}_4)_2\text{S}$; the mixtures were heated for thirty minutes in a covered casserole, 2 m.grms. W as Na_2WO_4 were added to each, and each was acidified with H_2SO_4 (1.20); to one mixture 3 c.c. 20 per cent FeCl_3 solution was at once added, and both mixtures were shaken and filtered; from that to which no FeCl_3 was added, a small yellowish pink precipitate separated; while in that to which it was added, a decidedly larger orange-yellow precipitate formed; the filtrate was distinctly yellow in both cases.

G. D., § 10.—300 m.grms. W as Na_2WO_4 , 300 m.grms. As as H_3AsO_4 , 100 m.grms. V as Na_3VO_4 , and 50 m.grms. Mo as $(\text{NH}_4)_2\text{MoO}_4$, in separate experiments, were digested with 10 c.c. $(\text{NH}_4)_2\text{S}$, the solution precipitated by H_2SO_4 ; the precipitate, sucked as dry as possible, was transferred to a stoppered Erlenmeyer flask, 20 c.c. HCl (1.20) poured over it, and the mixture heated for twenty minutes on a steam-bath; the flask was connected by means of tubes passing through the stopper with another flask containing a solution of $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$ strongly alkaline with NaOH, and at the end of the twenty minutes it was removed from the steam-bath and a current of air was drawn through the liquid so as to carry the vapours above it into the $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)$ solution; in the cases of the tungsten, arsenic, and vanadium, black precipitates, estimated at 10 m.grms., 5 m.grms., and 15 m.grms. respectively, separated, while in the case of the molybdenum not even a darkening was produced. The acid solution resulting from the action of HCl (1.20) on the WS_3 in this experiment was decanted

* From the *Technology Quarterly*, xvii., No. 3.

through a filter and evaporated to dryness; 10 c.c. HNO_3 (1·20) were added, and the mixture heated; a yellow residue estimated at 10 m.grms. remained, much of which had already separated during the evaporation of the HCl .

The experiment was repeated with 300 m.grms. W , except that HCl (1·12) was used in place of the HCl (1·20); the precipitate of PbS produced seemed to be equal in quantity to that obtained in the preceding test.

300 m.grms. W obtained as WS_3 , as described above, were heated in a flask on a steam-bath for thirty minutes with 20 c.c. HCl (1·20), a current of H_2S being passed continuously through the liquid; the solution was filtered and evaporated to dryness, and HNO_3 was added; there was a yellow residue not much less in quantity than that obtained when no H_2S was passed in.

(To be continued).

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, May 3rd, 1906.

Prof. H. E. ARMSTRONG, F.R.S., Past-President, in the Chair.

MESSRS. H. C. Irving and W. H. Glover were formally admitted Fellows of the Society.

The CHAIRMAN gave expression to the sense of loss sustained by the Chemical Society in the death of Professor Pierre Curie, which followed an accident on April 19th. The meeting endorsed the letter of condolence addressed by the President to M^{me}. Marie Curie, an Honorary and Foreign Member of the Society.

Certificates were read for the first time in favour of Messrs. Arthur John Berry, 5, University Gardens, Glasgow; William John Bowis, Ph.D., 97, North Road, West Bridgford, Notts; Harold Calam, B.Sc., 71, Cemetery Road, Beeston Hill, Leeds; John Denton, Horton Villa, Bradford; Bertie James Eaton, Kuala Lumpur, Federated Malay States; Arthur Burton Shepherd, B.Sc., 18, Stafford Street, Hull; Fred Smith, Cliff House, Bruntcliffe, near Leeds; Henry Wren, 22, Stapleton Hall Road, Stroud Green, N.; George Young, B.A., B.Sc., 38, Havelock Street, Sheffield.

A ballot for the election of Fellows was held, and the following were subsequently declared duly elected:—Ernest Barrett, B.Sc.; Richard Henry Beckett, B.Sc.; Roderick Harold Capper Birt, B.A.; Percy Garratt Chamberlain, M.A.; Edwin Rodney Chrystall; Herbert Reginald Cooper; Charles Davidson; William Dickson; Robert K. Duncan; Ernest Feilmann, B.Sc., Ph.D.; James Louis Foucar, B.Sc.; P. Bertram Foy; Edward Gardner; Richard Godfrey Hamilton Garvey; Hem Chandra Dutt Gupta, M.A.; Richard John Hall, M.Sc.; James Stuart Hills; William Tabor Lattey; Hamilton McCombie, M.A., B.Sc., Ph.D.; Ram Chandra Mukerjee, B.A.; Thomas Jenkins Murray, Ph.D.; Edgar Gale Oliver, M.A.; Ernest Ormerod, M.Sc., Ph.D.; Lawrence George Richardson; Walter Daly Scouller, B.Sc.; David Sommerville, B.A., M.D.; James Frederick Spencer, M.Sc., Ph.D.; Foster Sproston, B.Sc.; Francis W. Storey, B.Sc.; Harold Augustine Tempny, B.Sc.; George Augustus Turner; John Adam Watson; John Henry West; Henry John Wiffen; Cecil Wyatt-Edgell, B.A.

Of the following papers, those marked * were read:—

*87. "The Relation between Absorption Spectra and Chemical Constitution. Part V. The Isonitroso-compounds." By EDWARD CHARLES CYRIL BALY, EFFIE GWENDOLINE MARSDEN, and ALFRED WALTER STEWART.

In the previous papers it was shown that when two carbonyl groups are adjacent to one another in a com-

pound, the process of isorropesis is set up between the residual affinities of the oxygen atoms, with the result that an absorption band is produced in the visible blue region of the spectrum, and the compounds are coloured yellow. The principle was extended to the quinones and to the nitrophenols and nitroanilines. A similar condition exists in the isonitroso-compounds, and in the present paper the yellow colour of these substances in alkaline solution is explained. From observations of the absorption spectra of several isonitroso-compounds in neutral and alkaline solution, it is found that the free substances most probably

have the constitution $\begin{array}{c} \text{R} \cdot \text{C} \cdot \text{O} \\ | \\ \text{R} \cdot \text{CH} \cdot \text{NO} \end{array}$, but in presence of sodium hydroxide the hydrogen atom marked with a star is replaced by sodium, and becomes labile, so that a tautomeric process occurs thus—



Isoorropesis then takes place between the $>\text{C} \cdot \text{O}$ and $>\text{C} \cdot \text{N}$ —groups, the tautomeric process being the actuating mechanism. The compounds are therefore yellow in alkaline solution, owing to the presence of an absorption band in the visible blue region of the spectrum, which is due to the isorropesis. There is thus complete analogy between the isonitroso-bodies and quinonemonoxime. An analogous condition is shown to exist in the case of isonitrosocamphor.

DISCUSSION.

Dr. HEWITT drew attention to the way in which Mr. Baly had represented both nitroso- and isonitrosocamphor as equilibrium mixtures, one particular configuration of the molecule being common to both substances. If X be in equilibrium with Y , and Y in turn with Z , then X and Z must also be in equilibrium, and it should be impossible to isolate two different substances, one containing molecules of configurations X and Y , and the other molecules of configurations Y and Z in the case of rapid velocities of transformation.

Dr. LOWRY considered it very improbable that any of the colourless compounds to which Mr. Baly had referred possessed the true nitroso-formula $>\text{CH} \cdot \text{NO}$, since this group usually produces a blue or green coloration. He was surprised that no reference had been made to Beckmann's

isoxime-formula, $\begin{array}{c} \text{NH} \\ \diagup \\ >\text{C} \\ \diagdown \\ \text{O} \end{array}$; nitrogen-ethers derived from

the isoximes had been prepared in several cases, and nearly all the colour changes to which Mr. Baly had referred might be explained by the conversion of a coloured oxime into a colourless isoxime and *vice versa* (compare *B.A. Report*, Cambridge, 1904, p. 222). He strongly objected to the application of the term "tautomerism" to substances such as isonitrosocamphor and ethyl acetate; these substances were certainly not "tautomeric" in the sense of Laar's hypothesis, and it would be preferable to describe them in accordance with their actual behaviour as examples of labile or dynamic isomerism.

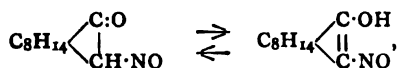
Mr. WM. ROBERTSON asked if Mr. Baly had examined the absorption spectra of the simple oximes. From the physical evidence, such as refractive indices, it would appear that the oximes and their salts were identical in structure, whereas in the case of the isonitroso-compounds a very different condition of affairs obtained. With regard to the isonitrosocamphors, he was of opinion that the stable isonitrosocamphor and its oxygen ethers contain the

grouping $\begin{array}{c} \text{C} \cdot \text{N} \cdot \text{OR} \\ || \\ \text{C} \cdot \text{O} \end{array}$, and that the salts derived from the unstable isomeride are represented by the grouping $\begin{array}{c} \text{C} \cdot \text{NOM} \\ | \\ \text{C} \cdot \text{O} \end{array}$.

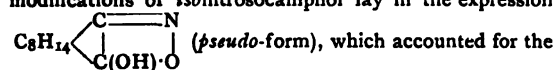
Dr. FORSTER stated that without having had an opportunity of examining the detailed communication, he had some difficulty in dealing with Mr. Baly's references to the views he had from time to time expressed regarding the constitution of *isonitrosocamphor*, but it seemed to him that the most important observation on that point which Mr. Baly and his colleagues had brought forward was the close similarity between the absorption spectra of the stable *isonitrosocamphor* and its O-alkyl ethers. Experiments of a merely chemical nature had made it clear that these ethers and the colourless benzoyl derivative are not

compounds of the type $\text{C}_8\text{H}_{14} \begin{array}{c} \text{C:N}\cdot\text{OX} \\ \text{C:O} \end{array}$, and the obvious

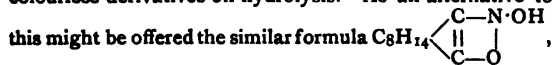
inference is that the stable *isonitrosocamphor* likewise belongs to a different class. Nevertheless, he was quite unable to accept the explanation of the authors, who, he understood, represented the stable modification as an equilibrium mixture,—



a system which should surely display some characteristics of a true nitroso-compound. If the stereochemical theory applied to *isonitrosocamphor* had to be abandoned, as to which the speaker was not completely convinced, a more satisfactory explanation of the difference between the two modifications of *isonitrosocamphor* lay in the expression



readiness with which α -camphornitrilic acid arose from the colourless derivatives on hydrolysis. As an alternative to



but, except in so far as it presented a smoother transition from the unstable form $\text{C}_8\text{H}_{14} \begin{array}{c} \text{C:N}\cdot\text{OH} \\ \text{C:O} \end{array}$, it pos-

essed no great advantage over the *pseudo*-form, although, in the speaker's opinion, certainly preferable to the isoxime constitution.

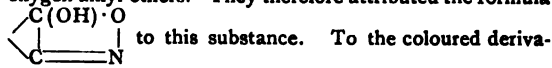
The CHAIRMAN congratulated the meeting on the fact that the paper had been so well discussed, and subjected to the proper criticism to which all papers presented to the Society should be open; it was clearly the general opinion that the conclusions arrived at were unsound. He could not agree that the word "isorropesis" coined by Mr. Baly was a good one, and he protested against its introduction; neither was it necessary, nor did it express the conception which had been brought forward. Apparently it indicated a process in which a state of equipoise or equilibrium was arrived at, to express which no new word was required; in reality, the idea put forward was that the production of absorption bands was due to a wobble or metamorphosis, to change of form, which also could be expressed without a new word. There was a growing practice among scientific workers, whenever a vague new conception was arrived at, to wrap it up in a new word which few could understand. He desired, with whatever authority the Chair gave, to protest in the most emphatic manner possible against the practice. He agreed with previous speakers that nitroso-formulae such as were put forward, could not be used for the colourless compounds under consideration; substances so constituted would be highly coloured. Mr. Baly objected to the interpretation put upon the term *isorropesis*, and denied that it meant wobble generally; it appeared that he limited it strictly to the wobble involved in the passage from the ketonic to the linked oxygen form of quinone.

Mr. BALY said, in reply to Dr. Hewitt, that the existence of one configuration being common to both processes was no objection. Two possibilities of labile isomerism exist

in *isonitrosocamphor*, and one of these they connected with the unstable variety and the other with the stable variety. The existence of a common configuration accounts for the ease with which unstable *isonitrosocamphor* passes over into the stable form.

In reply to Dr. Lowry, he thought that there was no direct evidence that the group $\text{>CH}\cdot\text{NO}$, as suggested, possessed a blue or green colour. The isoxime formula did not seem capable of explaining the absorption curves obtained with the *isonitroso*-compounds. In alkaline solution these bodies all show a strong absorption band in the ultra-violet analogous to ethyl acetoacetate in alkaline solution. Such a band as this does not seem capable of explanation by an equilibrium between the isoxime and oxime form; it must be due to the same type of labile isomerism as is occurring in ethyl acetoacetate.

In reply to Mr. Robertson, the absorption curves show that the stable form of *isonitrosocamphor* in neutral solution possesses the same configuration as Dr. Forster's oxygen alkyl ethers. They therefore attributed the formula

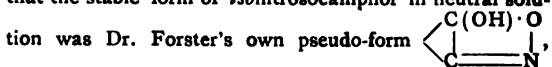


to this substance. To the coloured derivatives of the unstable form they attributed the formula

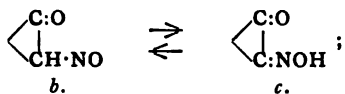


, but if the unstable form were colourless it could not have the formula $\begin{array}{c} \text{C:NOH} \\ \text{C:O} \end{array}$.

In reply to Dr. Forster, Mr. BALY apologised for not making himself clear upon the suggestions his colleagues and he brought forward. They were firmly of the opinion that the stable form of *isonitrosocamphor* in neutral solution was Dr. Forster's own *pseudo*-form $\begin{array}{c} \text{C(OH)\cdot O} \\ \text{C=N} \end{array}$,

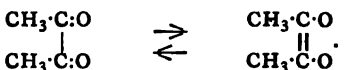


the colour in alkaline solution being due to the parallel linking in a. Similarly, in the unstable form, their view was that in alkaline solution the labile tautomerism could be best expressed by—

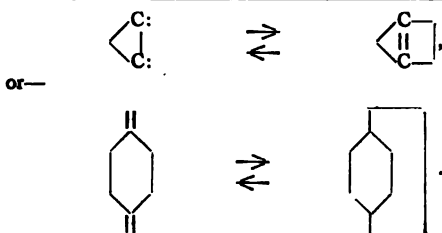


the colour was due to the *isorropesis* occurring with form c. It was difficult to give a formula to the unstable form in neutral solution, as its colour was not absolutely known.

There seemed to be confusion as to the meaning of the term *isorropesis*, and he would like to state what the process consists in. The simplest case is that of diacetyl, in which compound the residual affinities on the ketonic oxygen atoms disturb one another, with the result that an oscillation is set up which can, perhaps, be graphically expressed—



This process has been named *isorropesis*, and, in its broadest sense, it means an oscillation between the residual affinities of two or more adjacent atoms or groups of atoms, thus:—



The process might also be looked on as the result of the mutual interference between two systems, each possessing possibilities of labile isomerism, as, for example, the two $\text{CH}_3\cdot\text{CO}$ groups in diacetyl.

*88. "The Residual Affinity of Coumarin as shown by the Formation of Oxonium Salts." By GILBERT THOMAS MORGAN and FRANCES MARY GORE MICKLETHWAIT.

Coumarin dissolves much more readily in concentrated hydrochloric acid than in water, and the strongly acid solution, when treated with excess of chloroplatinic acid, yields a yellow crystalline *platinichloride*, $4\text{C}_9\text{H}_6\text{O}_2\cdot\text{H}_2\text{PtCl}_6\cdot 4\text{H}_2\text{O}$, which is dissociated into its generators by water. 6-Aminocoumarin, ethyl-6-aminocoumarin, dimethyl-6-aminocoumarin, and the corresponding quaternary base, like other aromatic amines, all yield *platinichlorides*, *platinibromides*, and *aurichlorides* of the normal type, but the acyl derivatives of 6-aminocoumarin, in which the salt-forming power of the nitrogen atom is largely destroyed, resemble coumarin itself in giving rise to *platinichlorides* containing a greater proportion of the organic constituent. *Acetyl-6-aminocoumarin platinichloride* has a composition approximating to the formula $4(\text{C}_7\text{H}_5\text{O}_2\cdot\text{NH}\cdot\text{CO}\cdot\text{CH}_3)\cdot\text{H}_2\text{PtCl}_6\cdot 4\text{H}_2\text{O}$.

Coumarin dissolves readily in warm fuming hydriodic acid containing free iodine, and from the dark brown solution, dark bronzy-green masses or well-defined needles separate, depending on the concentration; the product, which can be re-crystallised from concentrated hydriodic acid, has the composition of a *hydriodide periodide*, $4\text{C}_9\text{H}_6\text{O}_2\cdot\text{HI}\cdot\text{I}_3$.

The formation of these salt-like additive compounds of coumarin agrees with the results of earlier investigators. Ebert found that this lactone yielded an extremely unstable hydriobromide (*Annalen*, 1884, ccxxvi., 347), whilst W. H. Perkin, sen., described a dichloride and dibromide (*Annalen*, 1871, clvii., 116). Moreover, coumarin exhibits an amphoteric character in combining also with metallic oxides and hydroxides.

DISCUSSION.

Dr. CAIN referred to his paper on "The Constitution of the Ammonium Compounds" (*Mem. Manchester Phil. Soc.*, 1904, xlviii., iii., No. 14), in which he had suggested formulæ for the so-called molecular compounds, similar to those now brought forward by Dr. Morgan. He did not believe in Werner's theory of "Haupt Valenz" and "Neben Valenz" as applied to the ammonium compounds, but was of the opinion that a satisfactory explanation was given by assuming the presence of triad halogen and tetrad oxygen.

Dr. HEWITT remarked that the formation of oxonium salts frequently coincided with the occurrence of another oxygen atom in the molecule, in addition to the salt-forming oxonium oxygen atom; not only in the case of pyrones and coumarins, but also with xanthone and fluorane derivatives. Dr. Cain's formulation of ammonium chloride was open to the objection that it seemed incompatible with the known ionisation of ammonium salts in aqueous solution; a difficulty in no way diminished when one applied corresponding formulæ to the tetra-alkylammonium bases and their salts.

Dr. MORGAN, in reply to a question from Mr. F. H. CARR, stated that they had made many attempts to combine coumarin with metallic salts, such as mercuric iodide, but without success.

89. "Brazilin and Hæmatoxylin. Part VII. Some Derivatives of Brasilein." By PAUL ENGELS and WILLIAM HENRY PERKIN, jun.

Brazilein, $\text{C}_{16}\text{H}_{12}\text{O}_5$, is the colouring matter which is produced when brazilin, $\text{C}_{16}\text{H}_{14}\text{O}_5$, is oxidised in alkaline solution by means of air.

When brazilein is treated with a large excess of methyl sulphate and caustic potash, it is converted into *trimethyl-brazilein*, $\text{C}_{16}\text{H}_9(\text{OMe})_3\text{O}_5$, the formula of which was controlled by repeated analysis and the determination of the methoxy-groups by Zeisel's method.

This new orange-yellow, crystalline substance separates from solvents in two modifications, which melt at 160° and 178° respectively. It combines with formic acid, yielding *trimethylbrazilein formic acid*, which crystallises in brilliant garnet-coloured prisms, melts at 143° , and is decomposed into its components by treatment with alcohol. Trimethylbrazilein dissolves in concentrated sulphuric acid, yielding a crimson solution which soon becomes orange, and, when poured into water, deposits a brilliant vermilion, crystalline precipitate which appears to be *trimethylisobrazilein sulphate* (compare J. J. Hummel and A. G. Perkin, *Trans.*, 1882, xli., 9).

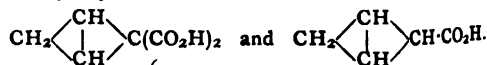
When trimethylbrazilein is left in contact with an alcoholic solution of hydroxylamine, it yields *trimethyl-brazilein hydroxylamine*, $\text{C}_{16}\text{H}_9(\text{OMe})_3\text{O}_5\cdot\text{NH}_2\cdot\text{OH}$, an almost colourless, crystalline substance which melts at about 155° with decomposition. This substance dissolves in dilute alkalis with a yellow colour, and is decomposed, on standing, into a neutral substance of melting-point 177° and trimethylbrazilein. It is also decomposed by boiling glacial acetic acid, and the solution, on cooling, deposits crystals of trimethylbrazilein.

The authors wish to reserve these new compounds for further investigation.

90. "The Action of Tribromopropane on the Sodium Derivative of Ethyl Malonate." By WILLIAM HENRY PERKIN, jun., and JOHN LIONEL SIMONSEN.

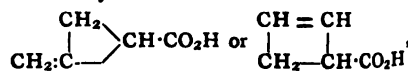
In a previous communication (*Proc.*, 1905, xxi., 256), it was shown that tribromopropane, $\text{CH}_2\text{Br}\cdot\text{CHBr}\cdot\text{CH}_2\text{Br}$, reacts with the sodium derivative of ethyl malonate with formation of an ester $\text{C}_{10}\text{H}_{15}\text{O}_2\text{Br}$.

This ester, when treated with alcoholic potash, yields an acid, $\text{C}_4\text{H}_4(\text{CO}_2\text{H})_2$, which is decomposed on heating, with elimination of carbon dioxide and formation of the corresponding monobasic acid, $\text{C}_4\text{H}_5(\text{CO}_2\text{H})$. Since these two acids do not decolorise bromine, are very slowly oxidised by permanganate, and are not affected by boiling with sodium amalgam, it was suggested that they are probably *dicyclobutane* derivatives of the formulæ—



These acids have now been prepared in considerable quantities, and the determination of the magnetic rotation of their esters has proved conclusively that they cannot have the above formulæ, but that they must be unsaturated closed chain compounds.

From a careful investigation of their behaviour, the authors incline to the view that the monobasic acid must be represented by one of the constitutional formulæ—



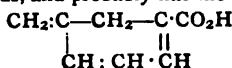
but such formulæ are, obviously, very difficult to establish.

Besides the bromo-ester mentioned above, a second *bromo-ester*, $\text{C}_{13}\text{H}_{18}\text{O}_4\text{Br}_2$, is formed during the action of tribromopropane on the sodium derivative of ethyl malonate; this distils at 191° (11 m.m.), and has either the constitution—

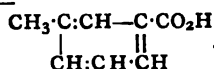


or some constitution closely allied to this.

When digested with alcoholic potash, it yields an acid of melting-point 47° , which closely resembles the *pseudo*-phenylacetic acids, and probably has the constitution—



This interesting acid instantly decolorises permanganate, and when boiled with water or mineral acids, or treated with acetic or hydrobromic acid in the cold, is converted quantitatively by molecular rearrangement into *m*-toluic acid (m. p. 110°)—



For this reason the acid of melting-point 47° has been named *pseudo-m-toluic acid*.

The further investigation of these new compounds is in progress.

91. "Pipitzahoic Acid." By JAMES MCCONNELL SANDERS.

An investigation of this substance was made by the author at the instance of the Mexican Government, with the object of correcting, if possible, the discrepancies which exist in the literature. Considerable attention is being attracted by this substance owing to its peculiar and valuable properties as a non-toxic purgative. The author describes the method of extraction from the Pipitzahoac root and the properties of the purified acid.

The formulæ of Weld ($\text{C}_{30}\text{H}_{20}\text{O}_6$) and Mylius ($\text{C}_{15}\text{H}_{20}\text{O}_3$) are rejected, the author considering that its composition is best represented by the formula $\text{C}_{10}\text{H}_{14}\text{O}_2$, it being thus isomeric with camphorquinone and similar to, although not identical with, the isocamphorquinone discovered by Manasse and formulated by Bredt as $\Delta^1-4(8)$ -terpadienol (*Ber.*, 1902, xxxvi., 3829—3843). Pipitzahoic acid is obtained in golden-yellow scales or orange crystals, and melts at $104-105^{\circ}$; by prolonged heating above its melting-point it loses carbon monoxide and carbon dioxide, giving at first a lemon-yellow sublimate, which melts at $88-89^{\circ}$, and finally a beautifully crystalline, colourless sublimate, which is insoluble in water, but soluble in most organic solvents. This compound, $\text{C}_{11}\text{H}_{14}\text{O}_2$, crystallises from acetone or benzene in glistening prisms melting at 140° , and having an odour of camphor. Its solutions are optically active, a 2 per cent solution in alcohol having $[\alpha]_D = -77.9^{\circ}$ at 25° ; it does not react with hydroxylamine or aniline, but with acetic anhydride forms a compound, $\text{C}_{41}\text{H}_{50}\text{O}_{10}$, which crystallises from acetone in square plates melting at $114-115^{\circ}$. Pipitzahoic acid is insoluble in water, but soluble in most organic solvents, its solutions being optically inactive. Its violet alkaline solution yields the unchanged substance on acidifying or by extraction with ether. It has a neutral reaction, does not contain methoxy-groups, and does not combine with sodium bisulphite. On fusion with potash, the acid gives methyl hexylene ketone and butyric acid. It forms an *oxime* (m. p. 153.7°), an aniline compound (m. p. $134-135^{\circ}$), and a *di bromide*, which is a yellowish brown, unstable oil. It is not reduced by sulphurous acid, but easily reduced by zinc dust and acetic acid, and when distilled in a current of hydrogen over zinc pumice gives a readily oxidisable liquid which forms optically inactive fluorescent solutions in chloroform and ether. The reduction product itself is isomeric with benzylidenecamphor, $\text{C}_{17}\text{H}_{20}\text{O}$. Pipitzahoic acid seems to behave as a hydroxy-ketone, forming a resinous acetyl compound and a greenish brown copper derivative, $(\text{C}_{10}\text{H}_{13}\text{O}_2)_2\text{Cu}$.

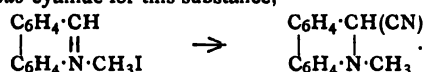
Silk may be dyed an olive-green tint by treating it with a solution of pipitzahoic acid after a preliminary mordanting with copper acetate.

92. "The Constitution of the Hydroxides and Cyanides obtained from Acridine, Methyl-acridine, and Phenanthridine Methiodides." By CHARLES KENNETH TINKLER.

From the results obtained by a study of the ultra-violet absorption spectra of the methiodides, before and after the

addition of caustic soda, it is concluded that the hydroxides produced are carbinols, and are constituted similarly to the hydro-compounds, as in the cases of cotarnine, hydrastinine, and phenylmethylacridol (Dobbie, Lauder, and Tinkler, *Trans.*, 1903, lxxxiii., 598; Dobbie and Tinkler, *Trans.*, 1904, lxxxv., 1005; and 1905, lxxxvii., 269).

The cyanide obtained from phenylacridine methiodide (Hantzsch, *Ber.*, 1899, xxxii., 3109) gives spectra almost identical with those of hydrophenylacridine, thus affording evidence in support of the constitution $\text{C}_6\text{H}_4\text{C}(\text{C}_6\text{H}_4)\text{CN}$ for this substance. A colourless cyanide has been prepared from phenanthridine methiodide. The absorption spectra of the substance agree very closely with those of hydrophenanthridine, and this is only to be explained by adopting the constitution of a *pseudo*-cyanide for this substance,—



93. "The Constitution of Ammonium Amalgam." By MISS ELIZABETH MARY RICH and MORRIS WILLIAM TRAVERS.

The authors have determined the freezing-points of a series of samples of ammonium amalgam, in which the concentration, calculated as grms. of ammonium per 100 grms. of mercury, varied between 0.0094 and 0.507. The value of the "molecular depression" agrees with that obtained by Tammann for solutions of other metals in mercury, leading to the conclusion that ammonium amalgam is a true solution of ammonium in mercury.

94. "Action of Light on Potassium Ferrocyanide." By GLYN WILLIAM ARNOLD FOSTER.

When a neutral or alkaline solution of potassium ferrocyanide is exposed to light, ferric hydrate is slowly precipitated; in presence of alkali sulphide, ferrous sulphide is thrown down.

No precipitation takes place if the solutions are protected from light; the action is therefore entirely photochemical. Potassium ferrocyanide is, in solution, dissociated in the usual manner, into potassium ions and the complex "ferrocyan" ions $[\text{Fe}(\text{CN})_6]^{4-}$; this is in absence of light. On exposure to light, this complex is dissociated into iron ions and cyan ions, and on removing the source of light the complex ferrocyan ion is regenerated. Thus no precipitation of iron can take place in absence of light.

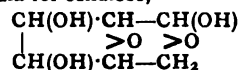
The relations $\text{Fe}:(\text{CN})$ were always below the theoretical 1:6, and this was found to be due to the oxidation of cyanide under the influence of light to cyanate and polymerides. In presence of alkali sulphide the cyanide is almost entirely converted into sulphocyanocompounds.

The source of light used was a mercury vapour lamp of quartz, made by W. C. Heraeus, of Hanau. It was water-cooled, and, using a direct current of 5 to 6 amperes at 240 to 250 volts, gave a very intense light, rich in ultra-violet radiation.

95. "Note on the Constitution of Cellulose." By ARTHUR GEORGE GREEN and ARTHUR GEORGE PERKIN.

It is generally believed that the highest acetyl derivative of cellulose is a tetra-acetate, $\text{C}_6\text{H}_6(\text{OAc})_4\text{O}$.

Green's formula for cellulose,—



(*Zeit. Farb. Text. Ind.*, 1904, iii., 97), contains only three hydroxyl groups, and would therefore yield a *tri*-acetate. As this formula affords a simple and direct explanation of the other reactions of cellulose, a re-examination of the acetate was desirable. Acetyl estimations of the so-called "tetra-acetylcellulose" were therefore made by three different methods:—(a) Perkin's method (*Trans.*, 1905, lxxxvii., 107); (b) alkaline saponification; (c) alkaline

saponification combined with Perkin's method. In each case the numbers obtained agreed closely with those required for the tri-acetate, $C_6H_7(OAc)_3O_2$.

The highest acetate, therefore, like the highest nitrate and benzoate, corresponds with the presence of three hydroxyl groups in the cellulose molecule.

It is suggested that the above expression represents cellulose in its simplest or unpolymerised form, but that fibre cellulose is a colloidal aggregate of a large number of such simple molecules.

96. "Some New Derivatives of Pinene." By FREDERICK PEACOCK LEACH.

When pinene nitroschloride is treated with potassium cyanate in alcohol at $50-60^\circ$, potassium chloride is precipitated, and on pouring the mixture into water a compound, $C_{12}H_{17}O_3N_3$, separates, which after crystallisation from alcohol melts at $238-240^\circ$. The substance is characterised by its great stability, being dissolved by hot concentrated nitric acid without undergoing change.

On reduction with zinc and acetic acid, carbon dioxide and ammonia are eliminated, the compound $C_{11}H_{18}ON_2$ being formed. This melts at 224° , and from its behaviour towards nitrous acid is most probably a ψ -carbamide, giving a well-crystallised nitroso-derivative melting at 159° ; when the latter is reduced, the corresponding ψ -semicarbazide is formed, yielding a benzal- ψ -semicarbazone, $C_{18}H_{23}ON_3$, which melts at 180° . When the compound $C_{12}H_{17}O_3N_3$ is heated with concentrated sulphuric acid, carbon dioxide and ammonia are eliminated, and there is produced a base, $C_{10}H_{18}ON_2$, which crystallises in needles and melts at $123-125^\circ$: $C_{12}H_{17}O_3N_3 + 2H_2O = C_{10}H_{18}ON_2 + 2CO_2 + NH_3$. This compound is amphoteric, and appears to be an amino-oxime; it is obtainable also from pinene nitroschloride by the action of ammonia.

97. "Glutaconic and Aconitic Acids." By SIEGFRIED RUHEMANN.

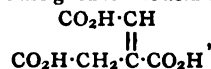
Rogerson and Thorpe show in their papers (*Trans.*, 1905, lxxvii., 1669, 1685; 1906, lxxxix., 631) on glutaconic and aconitic acids that they are unacquainted with several of my publications dealing with these acids, and they misrepresent some of my statements. I feel reluctantly obliged, therefore, to point out their errors.

After having found that ethyl aconitate, on treatment with ammonia, yields citrazinamide (*Ber.*, 1887, xx., 3366), I applied this reaction to the β -alkyl derivatives of ethyl glutaconate, and thus arrived at the β -alkyl substitution products of 2:6-dihydroxypyridine. Thus methyl, ethyl, and benzyl 2:6-dihydroxypyridine have been obtained (*Trans.*, 1893, lxiii., 259, 874), which are oxidised by ferric chloride to yellow substances. Lately, Rogerson and Thorpe have prepared derivatives of 2:6-dihydroxypyridine (*Trans.*, 1905, lxxvii., 1685) which contain alkyl groups in the γ -position and others with the same groups in the β - and γ -positions, and have pointed out that these derivatives react with ferric chloride in a manner different from the β -substituted 2:6-dihydroxypyridines (see Ruhemann, *loc. cit.*), but these chemists appear to be unaware of the fact that I had previously obtained derivatives of 2:6-dihydroxypyridine containing alkyl groups in the γ - and $\gamma\beta$ -positions by the action of ammonia on the products of the union of ethyl phenylpropionate with ethyl malonate or its homologues, and that I have also described their reaction with ferric chloride (*Trans.*, 1899, lxxv., 245). Again, in recording the properties of the $\alpha\alpha$ -dihydroxypyridines, Rogerson and Thorpe indicate the analogy which exists between these substances and resorcinol, but they seem to have overlooked the fact that I have already recorded this (*Ber.*, 1893, xxvi., 1559).

By the action of ethyl α -diocynoacetate on ethyl oxaloacetate, Rogerson and Thorpe (*Trans.*, 1906, lxxxix., 640) obtained ethyl α -cyanoaconitate which, under the influence of concentrated sulphuric acid, condenses to ethyl 2:6-dihydroxypyridine-4:5-dicarboxylate. They further show that, on boiling this ester with alkali, it is transformed into citrazinic acid. They state that ethyl

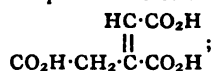
dihydroxycinchomerate had first been prepared by Errera and Perciabosco (*Ber.*, 1901, xxxiv., 3713), but they have overlooked the fact that Ruhemann and Stapleton (*Trans.*, 1900, lxxvii., 250) had obtained it a year before by the action of ammonia on ethyl propenetetracarboxylate, and had described its conversion into citrazinic acid.

I am especially surprised that Rogerson and Thorpe should have misunderstood Ruhemann and Orton's statements concerning the configuration of aconitic acid. They say (p. 634) that we assigned to this acid the configuration—



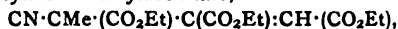
"on account of its formation from citrazinamide by the action of caustic potash at 150° , and also owing to the formation of citrazinamide by the action of ammonia on ethyl aconitate." As a matter of fact, the former reaction has never been advanced as a proof of the fumaroid structure. This was mainly derived from a careful study of the transformations of citrazinamide. I need not here enter into the arguments which support this view, because they are fully discussed in the paper to which Rogerson and Thorpe refer (Ruhemann and Orton, *Ber.*, 1894, xxvii., 3449).

The research described in this paper points to the existence of the other possible stereoisomeride,—

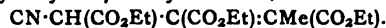


it was suggested only that the latter is "probably" identical with Baeyer's aconitic acid (*Annalen*, 1865, cxxxv., 306), but not put forward as our definite opinion, as Rogerson and Thorpe seem to imagine (*Trans.*, 1906, lxxxix., 634).

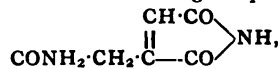
Rogerson and Thorpe arrive at a different conclusion as to the formula of aconitic acid by assigning to it a symmetrical structure, similar to that which they give to glutaconic acid. They support this view by demonstrating the formation of the same methylaconitic acid from both ethyl α -cyano- α -methylaconitate,—



and ethyl α -cyano- γ -methylaconitate,—



The same criticism which Feist and Beyer (*Annalen*, 1906, cccxlv., 117), who succeeded in proving the existence of both stereoisomeric β -methylglutaconic acids, offer to Rogerson and Thorpe's symmetrical structure of glutaconic acid applies also to the symmetrical structure of aconitic acid. Without entering into further discussion of the conclusions at which these chemists arrive, I will only point out that if their view were correct, the action of ammonia on ethyl aconitate may be expected to yield, besides citrazinamide, a five-membered ring compound,—



but the formation of this compound does not take place.

Research Fund.

A Meeting of the Research Fund Committee will be held in June next. Applications for grants, to be made on forms which can be obtained from the Assistant Secretary, must be received on or before Monday, June 4th, 1906.

Those Fellows who received grants in June, 1905, or whose grants, allotted in June of previous years, have not been closed, are reminded that reports must be in the hands of the Hon. Secretaries not later than Friday, June 1st, 1906.

Cleve Memorial Lectures.

The Cleve Memorial Lecture will be delivered by Prof. T. E. Thorpe, C.B., F.R.S., on Thursday, June 21st, 1906, at 8.30 p.m., before the business of the Ordinary Meeting.

PHYSICAL SOCIETY.

Ordinary Meeting, May 11th, 1906.

Dr. C. CHREE, F.R.S., Vice-President, in the Chair.

A PAPER ON "*The Dead Points of a Galvanometer Needle for Transient Currents*," was read by Mr. A. RUSSELL.

When many types of needle galvanometer are connected with a condenser and a battery in the ordinary manner by a charge and discharge key the following phenomena can easily be observed:—When the needle is initially at right angles to the axis of the galvanometer-coil, and the spot of light is in the centre, X, of the scale, the throws on charge and discharge are equal. If the controlling magnet be turned through a small angle, or if the suspending fibre be twisted slightly so that the spot of light is not in the centre of the scale initially, the throws on charge and discharge are not equal. The algebraic difference between them, however, is constant. Hence, for an initial position P_1 of the spot of light there is no throw on charge, and similarly for another initial position P_2 there is no throw on discharge. These points the author calls the dead points. If the resistance of the charging and discharging circuits are the same, $XP_1 = XP_2$. In this case the distance P_1P_2 varies directly as the resistance of the charging circuit, and inversely as the applied voltage. If the initial position of the spot of light be outside of P_1P_2 , the throws on charge and discharge are in the same direction. The author shows that these effects can be explained with considerable accuracy by supposing that the magnetism of the needle consists of two parts, one permanent and the other proportional to the magnetising force. As far back as 1868 Lord Rayleigh showed that this supposition is sufficient to explain the permanent deflexion produced by an alternating current passing through the galvanometer-coil. In a paper also, in 1883, he pointed out that a galvanometer is a very imperfect instrument for indicating whether the integral sum of the transient currents through it is zero or not, and he also mentioned the limitations this imposes on Maxwell's method of comparing mutual inductances. The author finds that it is easy to arrange with a low-resistance galvanometer so that a, relatively speaking, gigantic charge can be passed through the coil without producing any throw at all. He also finds that all the galvanometers he has tested, whether needle or moving coil, will produce throws when certain transient currents pass through them, even although the integral value of these currents is zero. It also appears that the effective internal resistance of ordinary condensers is appreciable in certain cases. These results are of importance in connection with several of the customary methods of testing.

Mr. W. DUDELL expressed his interest in the paper, and remarked that the phenomena described were of importance in the measurement of capacity. He suggested that since the R term in the paper included all the i^2R losses, part of it might arise from eddy-currents in the condenser. The condenser shown by the author was constructed by winding together very long strips of tin-foil, and the resistance should therefore appear high if it was true resistance; perhaps Mr. Russell could tell him the value of the resistance of the condenser shown.

Prof. H. A. WILSON, referring to the effective internal resistance of a condenser, pointed out that if there was dielectric hysteresis there must be a loss of energy, which no doubt amounted to a resistance as determined by the author.

Mr. ROLLO APPELYARD asked if it was necessary to assume that R was a resistance. It might be a physical constant of the condenser of another kind.

Mr. A. CAMPBELL said that the effects mentioned by Mr. Russell often gave trouble in actual testing-work. Some years ago he found that in testing standard air-condensers by Maxwell's method, an ordinary needle galvanometer gave erroneous results unless its needle was exactly in the symmetrical position. A moving coil-galvanometer, however, gave satisfactory results.

The AUTHOR, in reply to Prof. Wilson, stated that dielectric hysteresis had probably some connection with the internal resistance of the condenser, but the effect was small. The energy radiated into space was also small. Mr. Duddell's suggestion of eddy-currents might clear up any lack of uniformity in results obtained with very rapid oscillatory discharges. The author also showed how the resistance of a condenser could be measured in a minute or two by his method. The resistance of the actual condenser used in the experiments was found to be about 9 ohms.

Prof. H. A. WILSON exhibited a Lippmann Capillary Dynamo and Electromotor from the George the Third Museum of King's College.

Mr. W. DUDELL exhibited some Mechanical and Electrical Phenomena occurring in the Telephonic Transmission of Speech.

The apparatus is intended to demonstrate as curves on a screen the simultaneous movement of the microphone transmitter-diaphragm, the current flowing into the telephone line, the current received at the far end of the line, and the movement of the receiver-diaphragm when sounds or speech are being transmitted. The similarity of and the difference between these four curves can be examined by the aid of the apparatus, and the distortion and attenuation produced by the resistance, capacity, and self-induction of the line can be demonstrated, as well as the distortions produced by the diaphragms of transmitter and receiver. The characteristic shapes of the curves corresponding to the different vowel-sounds and their dependence on the pitch on which they are sung can also be exhibited.

CITY AND GUILDS OF LONDON INSTITUTE.

A GENERAL Meeting of Old Students was held at the Technical College, Finsbury, on Tuesday evening, May 8th, 1906, at which Sir OWEN ROBERTS, M.A., D.C.L., J.P., took the chair. About 120 old students attended this meeting to discuss the proposal to form an Old Students' Association. In a short opening speech Sir Owen Roberts expressed his approval of such Associations, and said that it gave him great pleasure to preside at the organisation meeting of such a one as this promises to be. Among other things he expressed his sympathy with its objects, and promised his hearty co-operation.

The CHAIRMAN then called on Mr. P. V. McMAHON, M.Inst.C.E., M.I.E.E., &c., to propose the following resolution:—"That an Old Students' Association be formed for—

- (a) The promotion of social intercourse;
- (b) The advancement of the interests of the Technical College and of past Students;
- (c) Mutual help."

This was seconded by Mr. T. J. UNDERHILL, Chief Inspector of Victualling Stores to H.M. Navy, and carried unanimously.

Mr. J. L. BAKER, F.I.C., Hon. Secretary of the London Section of the Society of Chemical Industry, then proposed, and Mr. C. E. HOLLAND, M.A., seconded, the next resolution, which was:—"That the draft scheme of the Organising Committee be provisionally adopted, and that it be an instruction to the Council to draw up a Constitution and By-laws, and to submit them to a General Meeting."

The following draft scheme was proposed:—

The Officers of the Association shall be a President, to be chosen annually in turn from each Department; Vice-Presidents, not to exceed six, of whom at least one shall be chosen from each Department; and three Secretaries, chosen by the Members of the Association.

Each Department shall elect a Standing Committee of nine Members to deal with purely departmental matters (e.g., the registration of members seeking or offering employment) who shall nominate five (of whom at least

one shall be an evening student) of their number to serve with the Officers as a General Council.

It is suggested that the appointment of a Standing Social Committee, and a Consulting Committee composed of Members of responsible position, to act with the Principal and Professors of the College, in advising, arbitrating, and helping Old Students in difficulties, should occupy the early attention of the Council.

All Past Students of the College shall be eligible as Members or Associates, but no one shall remain an Associate who has left the College more than five years. Past Members of the Staff, and Members of the Governing Body of the Institute, shall be eligible for full membership, and all present members of the staff shall be Honorary Members.

The subscription shall be 10s. 6d. per annum for Members, and 5s. for Associates.

After much discussion the resolution was passed with the following amendments:—(1) That there should be one class of Members only; (2) that the subscription be 5s. for London Members and 2s. 6d. for Country Members.

During the discussion Sir OWEN ROBERTS said that he felt sure that various City Companies would give financial support to the movement, and that he would put the matter before the Court of the Clothworkers' Company, which he represented.

The business of electing the Officers of the Association was then proceeded with, and Mr. A. H. MACCALLUM proposed, and Mr. R. G. PELLY, A.I.C., seconded, Dr. M. O. Forster, D.Sc., F.R.S., &c., as President of the Association. Dr. Forster's name was received with much enthusiasm, and he was unanimously elected.

The following officers were then proposed by Mr. MACCALLUM and seconded by Mr. PELLY, and were duly elected:—

Vice-Presidents—Sir Owen Roberts, M.A., D.C.L., J.P.; Prof. J. Perry, D.Sc., F.R.S., &c. *Mechanical*—C. G. Redfern. *Electrical*—P. V. McMahon, M.Inst.C.E., M.I.E.E., M.I.Mech.E. *Chemical*—H. D. Richmond, F.I.C.

Treasurer—Prof. W. E. Dalby, M.A., B.Sc., M.Inst.C.E., &c.

Secretaries.—*Mechanical*—S. E. Gritton. *Electrical*—E. W. Moss. *Chemical*—J. W. Brooker, A.I.C.

Five Members from each Department to serve on the Council:—

Electrical—A. C. Eborall, M.I.E.E.; H. W. Handcock, M.I.E.E.; H. B. Atkinson, M.I.E.E.; F. H. Halder, M.I.E.E.

Mechanical—A. H. MacCallum; H. N. Waynforth, A.M.I.M.E.; B. Chatterton; M. de Ville, A.M.I.M.E.

Chemical—J. L. Baker, F.I.C.; G. T. Morgan, D.Sc., F.I.C.; M. Priest, F.I.C.; W. J. Pope, F.R.S., F.I.C.; A. R. Ling, F.I.C.

The newly-elected PRESIDENT then proposed a vote of thanks to Sir Owen Roberts for kindly acting as Chairman. Prof. S. P. THOMPSON, D.Sc., F.R.S., &c., in seconding the vote, which was very heartily accorded, gave a short history of the movement, and said how pleased he was to see the Association thus formed.

MISCELLANEOUS.

Royal Institution. — On Tuesday next, May 29, at 5 o'clock, Colonel V. Balck begins a course of two lectures at the Royal Institution on "Northern Winter Sports—Sweden and its People"; and on Saturday, June 2, at 3 o'clock, Professor W. Macneile Dixon delivers a lecture on "The Origins of Poetry." The Friday Evening Discourse on June 1 will be delivered by Professor H. Moissan on "L'Ébullition des Métaux," and on June 8 by Professor Sir James Dewar on "Studies on Charcoal and Liquid Air."

NOTES AND QUERIES.

* Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Reviving Faded (Black) Ink.—(Reply to S. G. W. Mann).—The writer once revived some very ancient writing in ink that had been black by steeping the parchment document in a cold weak aqueous infusion of nut galls. For safety, only a small portion should be operated on at first.—SCRIBE.

MEETINGS FOR THE WEEK.

MONDAY, 28th.—Society of Arts, 8. (Cantor Lectures). "Heraldry in relation to the Applied Arts," by George W. Eve.

TUESDAY, 29th.—Royal Institution, 5. "Northern Winter Sports," by Col. V. Balck.
— Society of Arts, 8. "Glass Cutting," by Harry Powell.

THURSDAY, 31st.—Royal Institution, 5. "Man and the Glacial Period," by Prof. W. J. Sollas, F.R.S.

FRIDAY, JUNE 1st.—Royal Institution, 9. "L'Ébullition des Métaux," by Prof. H. Moissan, D.Sc., &c.

SATURDAY, 2nd.—Royal Institution, 3. "The Origins of Poetry," by Prof. W. Macneile Dixon, M.A., &c.

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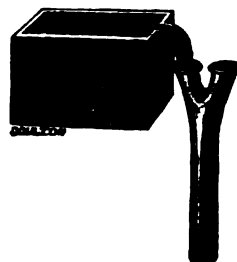
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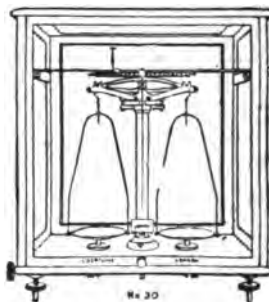
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THE CHEMICAL NEWS.

VOL. XCIII., No. 2427.

ON THE DISTRIBUTION OF RADIUM IN THE EARTH'S CRUST, AND ON THE EARTH'S INTERNAL HEAT.*

By the Hon. R. J. STRUTT, F.R.S., Fellow of Trinity College, Cambridge.

(Concluded from p. 237).

4. Results for Igneous Rocks.

THE results for igneous rocks will now be given in tabular form and in order of radium content. (See Table II.).

It will be observed that, in general, rocks like granite, with a high percentage of silica, are richer in radium than basic rocks. The rule, however, is by no means invariable.

Uranium ores occur in several of the rocks examined; thus the granites Nos. 2 and 6 occur near the pitchblende-bearing veins which were worked at Wheal Trenwith, St. Ives. The Norwegian syenite rocks, Nos. 3, 8, and 15, also contain local deposits of uranium-bearing minerals. These various rocks are fairly rich in radium, but do not stand in a class by themselves.

Thus the concentration of radium in such deposits is extremely local, and cannot appreciably disturb any general conclusions as to the total amount of radium in the earth's crust.

Confirmatory of this conclusion is the fact that the temperature gradient at Wheal Trenwith, St. Ives, is quite normal (see Prestwich, "Controverted Questions in Geology," p. 216). Considerable quantities of pitchblende occur in this mine, but they are evidently insufficient to cause appreciable disturbance in the distribution of temperature.

5. Meteorites.

Determinations have been made on one stony meteorite, on three samples of meteoric iron, and on a sample of native iron from Ovivak, Disco Island, Greenland (see Table III.). The quantities available have been various, and are entered on the list of results. Where the radium is entered as 0, it is to be understood that no leak was obtained which could clearly be distinguished from that normal to the electroscop.

It will be observed that the stony meteorite contains about as much radium as those basic terrestrial rocks which it resembles in general composition. No evidence was obtained of the presence of radium in iron meteorites. The Greenland iron contains a little radium. This was probably present in the siliceous material contained in this iron, which had been filtered off, decomposed by fusion, and added to the main solution.

6. The Earth's Internal Heat.

If q be the mean mass of radium per c.c. in the earth, h the heat production of radium per gm. per second, then the total heat production must be, per second,—

$$q \cdot h \cdot \frac{4}{3}\pi R^3.$$

If k be the thermal conductivity of the surface rocks, then the total outflow of heat per second will be—

$$4\pi R^2 k \cdot \left(\frac{d\theta}{dr}\right)_R;$$

where $\left(\frac{d\theta}{dr}\right)_R$ is the temperature gradient near the surface, as observed experimentally.

If the earth is in a thermally steady state, then these expressions will be equal, i.e.,—

$$4\pi R^2 k \left(\frac{d\theta}{dr}\right)_R = \frac{4}{3}\pi R^3 \cdot q \cdot h,$$

or—

$$q = \frac{3k}{hR} \left(\frac{d\theta}{dr}\right)_R.$$

For k I take the value 0.0041 (see Prestwich, *loc. cit.*). For the temperature gradient 1° F. in 42.4 feet (*loc. cit.*), or, in C.G.S. system, 4.3×10^{-4} .

One gm. of radium bromide produces 100 calories per hour. This gives for h the value 4.75×10^{-8} .

R , the earth's radius = 6.38×10^8 . Thus—

$$q = \frac{3 \times 4.1 \times 10^{-4} \times 4.3 \times 10^{-4}}{4.75 \times 10^{-8} \times 6.38 \times 10^8} = 1.75 \times 10^{-13}.$$

If therefore we assume the earth to be in thermal equilibrium, then, even if the whole of the internal heat is due to radium, the mean quantity per c.c. cannot much exceed 1.75×10^{-13} gm. per c.c., always supposing that the heat production of radium is not materially diminished under the conditions prevailing inside the earth.

Rutherford (*loc. cit.*), taking somewhat different values for the constants involved, gave the value 2.6×10^{-13} radium bromide, equivalent to 1.52×10^{-13} gm. radium per c.c.

It will be observed that all the igneous rocks examined, without exception, contain far more radium per c.c. than this. The poorest of all, Greenland basalt, contains more than ten times as much; an average rock something like fifty or sixty times.

The question must be faced:—Why has not the earth a temperature gradient far larger than that observed?

The calculation given above assumes:—

1. That the earth is in thermal equilibrium, i.e., that the amount of heat which escapes per second is equal to the supply produced in that time.
2. That no other source of internal heat than radium exists.
3. That a gm. of radium produces as much heat inside the earth as at the surface.

As to the first assumption, to suppose that the earth is cooling only aggravates the difficulty. To assume that it is getting hotter is an explanation not likely to be regarded with favour. I have not yet considered it quantitatively.

As to the second assumption, there can be little doubt that a quantity of uranium proportional to the amount of radium, exists in the rocks. (Too little to be detected in the ordinary course of analysis). Moreover, a trace of thorium is not improbably present. These sources of heat are not likely to be important, as compared with radium. There is, too, the possibility of the radio-activity of ordinary materials. If, however, a heating effect from them is assumed, of the order of magnitude to be expected from the ionisation they give, a temperature gradient would result something like 1000 times larger than that observed. I think this argument is conclusive against the theory that they have a genuine radio-activity of this order of magnitude. (See *Nature*, December 21, 1905).

There remains the third assumption, stated above. This cannot be passed over so lightly as the others, but will be more conveniently discussed later. I shall suppose for the moment that it is justified, and that the earth cannot contain more on the average than 1.75×10^{-13} gm. of radium per c.c.

For surface rocks the experiments show that 5×10^{-12} gm. per c.c. is a representative value. This is, if anything, an understatement. Thus not more than about 1/30 of the earth's volume can consist of material similar to that encountered on the surface. This gives a depth of about 45 miles for the rocky crust, assuming the total absence of radio-active material within.

* A Paper read before the Royal Society, April 5, 1906.

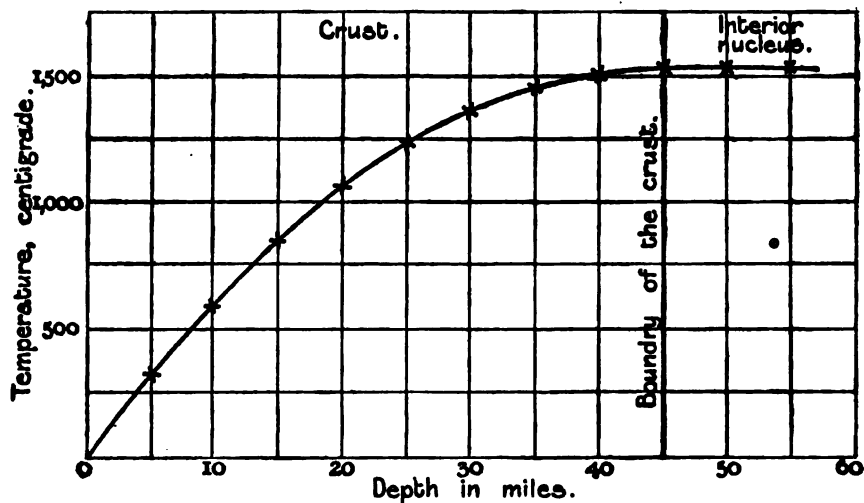


FIG. 2.—Calculated Distribution of Temperature in the Earth's Interior.

TABLE II.

No.	Name of rock.	Locality.	Density.	Radium per grm., in grms.	Radium per c.c., in grms.
1.	Granite	Rhodesia	2.63	9.56×10^{-12}	25.2×10^{-12}
2.	Granite	Lamorna Quarry, Cornwall	2.62	9.35	24.5
3.	Zircon syenite	Brevig, Norway	2.74	9.30	25.5
4.	Granite	Rosemorran, Cornwall (?)	2.62	8.43	22.1
5.	Granite	Cape of Good Hope	2.67	7.15	19.1
6.	Granite	Knill's Monument, near Carbis Bay, St. Ives, Cornwall	2.61	6.90	18.0
7.	Granite	Shap Fell, Westmoreland	2.65	6.63	17.6
8.	Elæolite syenite	Laurdal, Norway	2.70	4.88	13.2
9.	Granite	Haytor, Devonshire ?	2.61	3.69	9.64
10.	Blue ground	Kimberley	3.06	3.37	10.3
11.	Leucite basanite	Mt. Somma, Vesuvius	2.72	3.33	9.07
12.	Hornblende granite	Assouan, Egypt	2.64	2.45	6.47
13.	Pitchstone	Isle of Eigg	2.41	2.06	4.97
14.	Hornblende diorite	Schriesheim, near Heidelberg	2.89	1.98	5.73
15.	Augite syenite	Laurvig, Norway	2.73	1.86	5.07
16.	Peridotite	Isle of Rum	3.15	1.37	4.32
17.	Olivine basalt	Talisker Bay, Skye	2.89	1.32	3.82
18.	Olivine eucrite	Isle of Rum	2.97	1.28	3.80
19.	Basalt	Victoria Falls	2.75	1.26	3.46
20.	Hornblende granite	Mt. Sorrel, Leicestershire	2.71	1.25	3.38
21.	Dolerite	Isle of Canna	2.95	1.24	3.65
22.	Greenstone	Carrick Dû, St. Ives	2.99	1.14	3.41
23.	Basalt	Giant's Causeway, Antrim	2.80	1.03	2.89
24.	Serpentine	Cadgwith, Lizard	2.60	1.00	2.60
25.	Granite	Isle of Rum	2.61	0.723	1.89
26.	Olivine rock	Isle of Rum	3.22	0.676	2.18
27.	Dunite	L. Scaivig	3.34	0.664	2.22
28.	Basalt	Ovifak, Disco Island, Greenland	3.01	0.613	1.84

TABLE III.

Material.	Locality.	Quantity taken.	Radium per grm.
Stony meteorite	Dhurmshala	50 grms.	1.12×10^{-12}
Meteoric iron	Thunda	60 "	0
Meteoric iron	Augusta Co., Virginia	32 "	0
Meteoric iron	Santa Catarina	50 "	0
Native iron	Disco Island, Greenland	200 "	0.424×10^{-12}

I shall next consider the distribution of temperature in a crust of this thickness. The curvature of so thin a crust is relatively small, and may, without appreciable sacrifice of accuracy, be disregarded. Let x be the depth at any

point, measured from the outer surface, q the quantity of radium per c.c., h the heat developed in one second by 1 grm. of radium, θ the temperature at the depth x , and k the thermal conductivity.

Then the equation of conduction is—

$$\frac{d\theta}{dx} = \frac{-qh}{k},$$

which gives by integration—

$$\theta = \frac{-qh}{2k}(x^2 + ax + b);$$

where a and b are constants.

If the surface of the earth be assumed to be at 0°C ., then $\theta = 0$ when $x = 0$.

If t be the thickness of the crust, $d\theta/dx = 0$, when $x = t$. This is clear, since the temperature must be a maximum at the inner boundary of the crust. The interior core, free from radium, must be at a uniform temperature throughout.

Substituting these values, we get—

$$b = 0 \text{ and } a = 2t.$$

Hence—

$$\theta = \frac{qh}{2k}(2tx - x^2).$$

Substituting the numerical values adopted in this paper,—

$$\theta = \frac{5 \times 10^{-12} \times 4.75 \times 10^{-10} \times (2 \times 7.25 \times 10^6 - x)}{2 \times 4.7 \times 10^{-11}} \\ = 2.90 \times 10^{-11} x (1.45 \times 10^7 - x).$$

The curve appended shows this distribution of temperature graphically.

The maximum temperature at the bottom of the crust will be where $x = 7.25 \times 10^6$. This gives $\theta = 2.90 \times 10^{-11} \times (7.25)^2 \times 10^{12} = 1530^\circ$, a temperature considerably below the melting-point of platinum.

(NOTE.—No doubt the assumption of constant conductivity at all temperatures is an unsatisfactory feature in this calculation. It is difficult to know what other assumption to make, however. Some experiments of Lord Kelvin and Mr. Erskine Murray (*Proc. Roy. Soc.*, vol. lviii., p. 162) seem to indicate a diminution of conductivity with temperature. On the other hand, Mr. C. H. Lees found the thermal conductivity of window glass to increase with temperature, and at high temperatures rock magmas must approach the quality of glass; indeed, they sometimes even retain that quality on cooling (obsidian and pitch-stone). The mean thermal conductivity of rock cannot be much more than that assumed, for otherwise the internal temperature would not be high enough to produce volcanic phenomena).

This result has been obtained on the provisional assumption that the heat production of radium is the same throughout the earth's crust as under surface conditions. In justification of this, a paper by Mr. W. Makower may be referred to (*Proc. Roy. Soc.*, A, vol. lxxvii., p. 241). The activity of radium emanation and its products are shown to be substantially the same at temperatures of 1200° as at the ordinary temperature, though evidence was obtained of a slight change of activity in one of the products. Thus there is no reason at present to think that notable change of activity sets in before 1500° is reached. I wish to express myself with some reserve on this subject. Further experiments might conceivably show a rapid loss of activity as this temperature was approached. In that case the conclusions here drawn as to the earth's internal condition would require modification.

I was inclined at first to think it incredible that the earth's crust could have so small a thickness as 45 miles, and was therefore much interested to hear that Professor Milne had come to a substantially identical conclusion, from a study of the velocity of propagation of earthquakes through the earth's interior (Bakerian Lecture, 1906). He gives 30 miles as the thickness. This is quite consistent with my data, if rocks like granite, rich in radium, are assumed to be somewhat more abundant than I have supposed, in taking the value 5×10^{-12} grm. radium per c.c. as representative.

Professor Milne expresses the opinion that a fairly abrupt transition occurs at a depth of 30 miles, and that the material below that depth is fairly uniform throughout the globe. This is entirely in agreement with the view put forward above with regard to the earth's interior.

The chemical nature of the interior is a difficult problem. It can scarcely consist mainly of iron, as has been very commonly supposed, from the analogy of meteorites. Meteoric iron is remarkably free from radium, as shown above, and in this respect answers the requirements well enough. But if the stony exterior of the earth is but a small fraction of the whole volume, it cannot have much influence on the mean density, which should be nearly equal to that of the core. The density of the earth (5.5), is much less than the density of iron (7.7).

7. Internal Heat of the Moon.

The data of this paper have an interesting application to the moon. What we can observe of the moon's surface suggests that it consists of rock like that on the earth. The moon is believed to have originally separated from the earth's surface, and should therefore consist of the same material as the latter. Moreover, the density of the moon (3.5) does not differ much from that of rock. It seems reasonable to conclude from these facts that the moon consists almost entirely of rock similar to that of the earth's crust.

On this view, the temperature gradient of the moon should be very great in comparison with that of the earth. The material of the moon is taken to be some thirty times richer in radium than the (mean) material of the earth. Her volume is about one-fiftieth that of the earth. Thus the total heat production in the moon would be about half of that in the earth. This heat has to flow out through about one-sixteenth the area of the earth surface. Thus the temperature gradient at the moon's surface should be eight times greater than at the earth's.

In addition to this, gravity is very much less on the moon. We may conclude that the conditions which prevail there are far more favourable to manifestation of the internal heat by volcanic upheaval. This fully explains why volcanic features are so much more prominent on the moon than on the earth.

It has generally been supposed that the lunar craters are extinct. But that view seems to rest chiefly on an *a priori* conviction that the moon has no internal heat. As Professor W. H. Pickering has pointed out, all those observers who have made a special study of the moon have believed in the reality of changes occurring there (*Nature*, January 5, 1905).

8. Summary of Conclusions.

1. Radium can easily be detected in all igneous rocks. Granites, as a rule, contain most radium, basic rocks the least.

2. This distribution of radium is uniform enough to enable a fair estimate to be made of the total quantity in each mile of depth of the crust.

3. The result indicates that the crust cannot be much more than 45 miles deep, for otherwise the outflow of heat would be greater than is observed to be the case. The interior must consist of some totally different material. This agrees entirely with Professor Milne's conclusion drawn from a study of the velocity of propagation of earthquake shocks through the interior.

4. The moon probably consists for the most part of rock, and if so, its internal temperature must be far greater than that of the earth. This explains the great development of volcanoes on the moon.

5. Iron meteorites contain little, if any, radium. Stony ones contain about as much as the terrestrial rocks which they resemble.

In conclusion, I must thank Mr. A. Harker, Mr. Clement Reid, and Mr. A. Hutchinson for their kindness in providing me with various specimens of rock, and for information on geological matters.

A SYSTEM OF QUALITATIVE ANALYSIS INCLUDING NEARLY ALL THE METALLIC ELEMENTS.*

PART II.—ANALYSIS OF THE TUNGSTEN GROUP.

By ARTHUR A. NOYES.

(Continued from p. 240).

Confirmatory Experiments and References (continued).

P. 31, N. 2.—One m.grm. Sb as Sb_2O_3 , 1 m.grm. Cu as CuSO_4 , and 1 m.grm. Bi as $\text{Bi}(\text{NO}_3)_3$ were separately dissolved in 5 c.c. 48 per cent HF, and 40 c.c. water, and H_2S was passed into the solution in a covered dish for fifteen minutes; a considerable precipitate formed in each solution, which was orange-coloured in the first case and black in the other cases.

100 m.grms. Bi as $\text{Bi}(\text{NO}_3)_3$ were dissolved in 5 c.c. 48 per cent HF and 40 c.c. water, the solution filtered, the filtrate saturated with H_2S , the precipitate filtered out, the filtrate evaporated with H_2SO_4 , and NH_4OH added; no precipitate resulted.

100 m.grms. Se as SeO_2 and 100 m.grms. Te as TeO_2 were separately dissolved in 3 c.c. 48 per cent HF and 24 c.c. water, and H_2S passed into saturation; a large precipitate immediately separated. This was filtered off, the filtrate evaporated to dryness, HCl (1·02) added, and H_2S passed first into the solution in the cold and then into it at a nearly boiling temperature; no more precipitate separated in either case.

500 m.grms. As_2O_3 were treated with HNO_3 (1·42), and the solution evaporated just to dryness; then 3 c.c. 48 per cent HF and 24 c.c. water were added, and H_2S passed in; a precipitate formed only after several minutes, and this continued to increase for an hour. It was then filtered out, and H_2S passed into the filtrate; a large precipitate again resulted.

100 m.grms. Mo as $(\text{NH}_4)_2\text{MoO}_4$ and 4 m.grms. W as Na_2WO_4 were dissolved in 5 c.c. 48 per cent HF and 40 c.c. water, and H_2S was passed into the cold solution for thirty minutes. The precipitate was treated by P. 43; there was only a scarcely noticeable yellow residue (of H_2WO_4) after the evaporation with H_2SO_4 . The filtrate from the H_2S precipitate was treated by P. 33, 35, 39, 41, omitting the HCl treatment, and P. 43; a fairly large yellow residue (of H_2WO_4) resulted.

A mixture of 400 m.grms. Mo as $(\text{NH}_4)_2\text{MoO}_4$, 8 m.grms. Sn, and 8 m.grms. W in HF solution was dissolved in 5 c.c. 48 per cent HF and 40 c.c. water; 2 c.c. H_2SO_4 (1·84) were added, and H_2S was passed for thirty minutes into the cold solution; the precipitate was treated by P. 41, 42, and 43, and the filtrate by the usual complete procedure; the HgCl_2 test obtained by treating the precipitate was only about one-fourth as large as that obtained by treating the filtrate; no tungstic acid separated in the analysis of the precipitate, while a large quantity was obtained in that of the filtrate upon treating the ignited sulphide with HNO_3 and H_2SO_4 .

300 m.grms. Mo as $(\text{NH}_4)_2\text{MoO}_4$ were dissolved in 5 c.c. 48 per cent HF and 40 c.c. water, H_2S passed into the cold solution for thirty minutes, and the mixture filtered; a large black precipitate resulted, and the filtrate became very turbid on short standing. The filtrate was placed on a steam-bath and H_2S passed into the hot solution for thirty minutes more, and filtered; a much smaller precipitate was obtained than before. This last precipitate was treated with HNO_3 (1·20), and the solution was filtered; the residue was ignited, and the filtrate from it evaporated, the two residues united, heated to a low red heat, and weighed; 39 m.grms. of MoO_3 corresponding to 26 m.grms. Mo were obtained. The filtrate from the second H_2S precipitate was evaporated, and the residue ignited and weighed; 45 m.grms. MoO_3 corresponding to 30 m.grms. Mo were obtained. This shows that of the

300 m.grms present only 56 m.grms. remained unprecipitated after the first thirty minutes' treatment with H_2S in the cold.

The last experiment was repeated, except that 3 c.c. H_2SO_4 were added to the HF solution before passing in H_2S , and except that no quantitative determinations were made, the two precipitates obtained were of nearly the same size as before.

500 m.grms. Sn as metal were treated with HNO_3 (1·42), and the latter evaporated off; the residue was dissolved in 5 c.c. 48 per cent HF and 40 c.c. water, and H_2S passed into the cold solution for over an hour; no precipitate appeared.

500 m.grms. W as Na_2WO_4 were dissolved in 3 c.c. HF and 24 c.c. water, and the cold solution was treated with H_2S gas for an hour; no precipitate separated.

A mixture of 500 m.grms. Sb and 2 m.grms. Sn as metals was treated with HNO_3 , the mixture evaporated, the residue dissolved in 48 per cent HF, the solution diluted with eight times its volume of water, and treated with H_2S gas for thirty minutes; the precipitate next filtered out, the filtrate evaporated with 3 c.c. H_2SO_4 , and the solution diluted and treated with granulated zinc and a piece of platinum, as long as gas evolution continued; then to the mixture strong HCl was added, and heat applied till the zinc was all dissolved, and the solution was filtered and added to HgCl_2 ; a small white precipitate, which turned black on treatment with NH_4OH , resulted. The experiment was repeated with 500 m.grms. Sb and 1 m.grm. Sn; no precipitate of HgCl_2 was obtained.

Five c.c. 48 per cent HF and 40 c.c. water were saturated cold with H_2S and kept so, while portions of 1 m.grm. Pb as $\text{Pb}(\text{NO}_3)_2$ were added till a permanent precipitate resulted; 6 m.grms. Pb were required.

P. 31, N. 3.—One m.grm. Sb as Sb_2O_3 was dissolved in 5 c.c. 48 per cent HF and 25 c.c. water, and H_2S passed in at the room temperature for thirty minutes; no precipitate formed. The solution was then cooled in ice water, and H_2S again passed; a distinct precipitate separated. The mixture was heated till the precipitate re-dissolved, then cooled to room temperature, again saturated with H_2S (whereby no precipitate was produced), diluted with 15 c.c. water, and again saturated with the gas; a distinct precipitate resulted.

500 m.grms. Sb as metal were treated with HNO_3 , the acid evaporated, the residue dissolved in 5 c.c. 48 per cent HF and 40 c.c. cold water, H_2S passed in half-an-hour, the precipitate filtered out, the filtrate evaporated to fuming with 1 c.c. H_2SO_4 , diluted with water (whereupon a large white precipitate separated), and H_2S passed into saturation; an orange precipitate not very different in quantity from that that separated from the HF solution was obtained.

A solution of 500 m.grms. Sn as SnO_2 and 1 m.grm. Sb in 5 c.c. 48 per cent HF and 40 c.c. water was saturated with H_2S in the cold; a decided orange-coloured precipitate separated.

One m.grm. Sb was dissolved in a mixture of 5 c.c. 48 per cent HF, 40 c.c. water, and 1 c.c. H_2SO_4 (1·84), and H_2S was passed in to saturation in the cold; no precipitate formed. The experiment was repeated with 2 m.grms. Sb; a distinct, compact, orange precipitate separated.

Two m.grms. As as As_2O_3 were dissolved in 5 c.c. 48 per cent HF and 40 c.c. water, and H_2S passed into the cold solution for fifty minutes; no precipitate whatever separated. The solution was then heated on a steam-bath, and H_2S again passed in; a precipitate appeared almost immediately.

A mixture of 400 m.grms. Mo as $(\text{NH}_4)_2\text{MoO}_4$, 2 m.grms. Ta, 2 m.grms. Ti, 2 m.grms. Sn, and 4 m.grms. W, dissolved in 5 c.c. 48 per cent HF and 40 c.c. water, was treated with H_2S , first cold and then hot; the solution became very dark coloured, but very little precipitate separated. Then 3 c.c. H_2SO_4 (1·84) were added; a very bulky, slimy precipitate immediately separated, causing the solution almost to gelatinise. The mixture was filtered;

* From the *Technology Quarterly*, xvii., No. 3.

the liquid ran through with extreme slowness. The filtrate was tested by the complete procedure for all of the elements present in small quantity; no test for any of them except tantalum was obtained, indicating that they were carried down by the slimy precipitate.

P. 33, N. 2.—100 m.grms. dehydrated H_2SiO_3 were dissolved in HF, H_2SO_4 was added, and the solution was evaporated to complete dryness; no residue could be seen, showing that one evaporation with HF and H_2SO_4 suffices to expel all the silica.

P. 33, N. 4.—500 m.grms. Sn as H_2SnO_3 , 300 m.grms. Ti as H_2TiO_3 , 100 m.grms. Nb as Nb_2O_5 , 100 m.grms. Ta as Ta_2O_5 , and 500 m.grms. Mo as MoO_3 were dissolved in separate experiments in a little HF, 2–3 c.c. H_2SO_4 (1·84) were added, the solution was evaporated till it fumed strongly, and then after cooling it was poured gradually into 5 c.c. cold water; no permanent precipitate separated in any case; the molybdenum solution had a deep blue colour, and before dilution there were blue spots above the surface of the liquid. The experiment was repeated with 100 m.grms. Nb, and with 100 m.grms. Ta, taking no especial care to cool the H_2SO_4 upon dilution; a large white flocculent precipitate separated in each case.

To 2 m.grms. W were added 2 c.c. HF, 0·5 c.c. HNO_3 , and 3 c.c. H_2SO_4 ; the solution was heated gently until most of the HF was expelled; it was then heated at 110° in a hot closet for thirty minutes; and finally it was added to 8 c.c. water; a precipitate separated almost immediately.

The experiment was repeated, except that the mixture was heated on a sand-bath just below the fuming-point for thirty minutes; upon dilution a precipitate began to separate.

To 2 m.grms. W and 4 m.grms. W as Na_2WO_4 , 2 c.c. H_2SO_4 (1·84) were added, and the solution evaporated to the fuming-point; even after cooling, the solution remained clear. The liquid was added to 5 c.c. water; a white turbidity appeared in three to five minutes in the 4 m.grms. portion, and in thirty minutes in the 2 m.grms. portion; on standing over-night, yellow precipitates had settled out in both cases. The experiment, with some variations, was many times repeated with 1 m.grm. W; in no case did any precipitate separate, even on standing.

100 m.grms. W as Na_2WO_4 were dissolved in a little water, 1 c.c. H_2SO_4 (1·84) added, and the mixture evaporated to the fuming-point; a large quantity of a heavy yellow powder separated.

P. 34, N. 3.—0·1 m.grm. PO_4 as Na_2HPO_4 was added to 5 c.c. of the $(\text{NH}_4)_2\text{MoO}_4$ reagent, and the mixture was warmed; a considerable yellow precipitate formed at once.

P. 35, N. 2.—To 300 m.grms. Mo as $(\text{NH}_4)_2\text{MoO}_4$, 300 m.grms. W as Na_2WO_4 , and 300 m.grms. V as Na_3VO_4 in separate experiments, 10 c.c. water, 3 c.c. NH_4OH , and 10 c.c. $(\text{NH}_4)_2\text{S}$ were added, and the mixture kept at 50 – 60° for forty-five minutes; everything stayed in solution in the case of the molybdenum and tungsten; a rather small green precipitate remained in the case of the vanadium; the molybdenum had a deep orange-red colour, the vanadium a yellow-brown colour. The experiment was repeated with 1 m.grm. Mo; a distinct orange colour was apparent.

Three portions of 500 m.grms. Sn as metal were treated with three portions of 5 c.c. HNO_3 (1·42), to one of which had been added 500 m.grms. V as Na_3VO_4 , to another 500 m.grms. W as Na_2WO_4 , and to the third 500 m.grms. Mo as $(\text{NH}_4)_2\text{MoO}_4$. The mixture was evaporated to dryness, the residue heated for one hour at 120° , and then treated by P. 5, 6, 33, and 35; on adding an excess of NH_4OH , nothing remained undissolved in the mixture containing vanadium, there was a yellowish white precipitate in that containing tungsten, and a greyish blue one in that containing molybdenum. On adding 10 c.c. cold $(\text{NH}_4)_2\text{S}$, the mixtures containing vanadium and molybdenum became deep red, and no precipitate remained; in that containing tungsten, everything also dissolved.

A solution of 300 m.grms. Ti and 5 m.grms. W in HF was treated by P. 33, 35, 36, and the first paragraph of

P. 38; a decided white precipitate was produced in the HgCl_2 solution, showing the presence of tungsten in the well washed $(\text{NH}_4)_2\text{S}$ precipitate. The experiment was repeated with many other mixtures of titanium and tungsten, with the same result.

As to the impossibility of completely removing tin and tungsten from niobium and tantalum with $(\text{NH}_4)_2\text{S}$, see Hall, "Observations on the Metallic Acids," thesis submitted to University of Pennsylvania, 1904, p. 9.

A mixture of 400 m.grms. Ti and 200 m.grms. Mo was dissolved in HF and treated by P. 33 and 35, the precipitate being three times washed with hot water upon the filter with the help of suction; it still had a strong orange colour. It was returned to a casserole, and heated with frequent stirring upon a water-bath with 10 c.c. NH_4OH (0·90) and 40 c.c. water; the precipitate was then filtered out and washed; it now had only a slight yellowish colour. It was dissolved in HF, and treated by the whole of P. 38; a large precipitate was obtained in the first HgCl_2 test, but none whatever in the second HgCl_2 test after the fusion with Na_2CO_3 and S, showing that the quantity of molybdenum retained in the $(\text{NH}_4)_2\text{S}$ precipitate after digestion with NH_4OH is not so great that it cannot be removed by the fusion. The experiment was repeated, except that the $(\text{NH}_4)_2\text{S}$ precipitate was only well washed with water, not digested with NH_4OH ; in this case a large precipitate was obtained in the HgCl_2 test, even after the fusion with Na_2CO_3 and S, showing that the digestion with NH_4OH may be necessary.

P. 36, N. 2.—100 m.grms. Ti as H_2TiO_3 and 100 m.grms. Nb as Nb_2O_5 , in separate experiments, were dissolved in HF and treated by P. 33, 35, and 36; the whole H_2TiO_3 precipitate dissolved in the H_2O_2 , and of the HfNbO_3 not more than 1 m.grm. remained undissolved. The experiment was several times repeated with 300 m.grms. Ti as H_2TiO_3 ; in every case a large white precipitate, amounting to from one-third to one-sixth of the original quantity, remained undissolved. This precipitate was treated successively with two fresh portions of H_2O_2 and HCl; each time the liquid was strongly coloured, but there was still a large residue. This residue was treated with 5 c.c. cold HF; it dissolved completely in two or three minutes.

Twenty m.grms. Ta as Ta_2O_5 were dissolved in HF, and the solution treated by P. 33, 35, and 36; the H_2O_2 solution was saturated with SO_2 and NH_4OH added; a precipitate about equal in amount to the residue undissolved by the H_2O_2 was produced. This residue was dissolved in HF, and again treated by P. 33, 35, and 36, and the H_2O_2 solution was saturated with SO_2 , and made alkaline with NH_4OH ; again a precipitate nearly equal in amount to the residue was obtained.

One m.grm. Ta as Ta_2O_5 in HF solution was treated by P. 33, 35, 36, and 37, and the H_2O_2 solution obtained in P. 36 was saturated with SO_2 , and made alkaline with NH_4OH ; about one-half the $(\text{NH}_4)_2\text{S}$ precipitate dissolved in the H_2O_2 , and from the residue a distinct precipitate of $\text{K}_2\text{TaF}_7 \cdot \text{Ta}_2\text{O}_5$ was obtained by P. 37.

300 m.grms. Sn and 300 m.grms. Ti in HF solution were treated by P. 33, 35, and 36; there was a large residue insoluble in H_2O_2 and HCl. This was removed from the filter and warmed with 48 per cent HF; a large residue still remained. This was fused with Na_2CO_3 and S, and treated with hot water; the mass dissolved completely, showing the absence of much titanium. The solution was tested for tin by P. 39, 41 (omitting the ignition), and 43; a very large precipitate of HgCl resulted.

P. 36, N. 3.—Two portions of 20 m.grms. Ti in HF solution were evaporated with 2 c.c. H_2SO_4 (1·84), the solution diluted with 5 c.c. water and a large excess of NH_4OH , and heated on a steam-bath for one hour; the precipitates were filtered off, washed, and transferred to test-tubes; to one of them 10 c.c. hot 3 per cent H_2O_2 , and to the other a hot mixture of 10 c.c. 3 per cent H_2O_2 and 2 c.c. HCl (1·12) were added, and the mixtures shaken for two minutes; in the first case, much of the precipitate

was undissolved, and the mixture had a light yellow colour; in the second case, a clear deep orange-red solution resulted.

P. 36, N. 4.—0.1 m.-gram. Ti and 100 m.-grms. Ti were added to 10 c.c. H_2O_2 and 2 c.c. HCl (1:12); a distinct yellow colour resulted in the first case, and a deep orange-red in the second.

P. 36, N. 5.—Five m.-grms. V and 20 m.-grms. V as Na_2VO_4 were added to 10 c.c. 3 per cent H_2O_2 and 2 c.c. HCl (1:12); an orange-yellow colour resulted in the first case and a deep orange-red in the second. The experiment was repeated with 5 m.-grms. W as Na_2WO_4 ; no colour was produced. It was repeated with 5 m.-grms. Mo and with 100 m.-grms. Mo as $(\text{NH}_4)_2\text{MoO}_4$; a very pale yellow colour, not so strong as that produced by 0.1 m.-gram. Ti, resulted in the first case, and a deep pure yellow colour in the second case.

P. 37, N. 2.—One m.-gram. Ta as Ta_2O_5 and 10 m.-grms. SiO_2 were dissolved in HF , and, omitting the usual evaporation with HNO_3 , K_2CO_3 was immediately added, the solution was evaporated, the residue heated and dissolved in a little water, and the solution boiled down to 1 c.c.; no precipitate separated, even on standing.

P. 37, N. 3.—For the solubility and composition of the potassium fluotantalate and fluoxytantalate, see Marignac, *Ann. Chim. Phys.*, 1866, (4), ix., pp. 268, 269.

A portion of 2 m.-grms. Ta was treated by P. 37, and two other portions of 2 m.-grms. Ta were treated in the same way except that the residue, after adding K_2CO_3 and evaporating, was in one case heated only till it became dry (but not enamel white), and was in the other case heated to redness; scarcely any precipitate formed in the portion heated only to dryness, a fairly large precipitate of the usual appearance separated in that treated exactly according to the procedure, and a lighter, more bulky, powdery one resulted in the portion heated to redness.

P. 37, N. 4.—For the solubility of K_2TiF_6 , see Marignac, *Ann. Chim. Phys.*, 1866, (4), viii., p. 65.

P. 37, N. 5.—100 m.-grms. Nb as HNbO_3 , precipitated from H_2SO_4 solution by NH_4OH , were dissolved in HF , and treated by P. 37; at the end a large granular precipitate separated in the cold when the volume was 1.5 c.c., but this dissolved on heating, and after 1 c.c. more of water was added, it remained in solution even after cooling.

The experiment was repeated with a mixture of 100 m.-grms. Nb and 1 m.-gram. Ta; at the end, when the volume was 1 c.c., the large granular precipitate separated as before, and this dissolved when 3 c.c. water were added, but there then remained a small quantity of the heavy, dirty white precipitate characteristic of tantalum.

For the solubility and composition of the potassium fluoxyniobate, see Marignac, *Ann. Chim. Phys.*, 1866, (4), viii., p. 28.

P. 38, N. 3.—Direct experiments confirmed all the statements in regard to the colours produced.

P. 38, N. 4.—Reduction of TiO_2 by zinc to Ti_2O_3 , see König and v. d. Pfordten *Ber. Chem. Ges.*, 1889, xxii., p. 2079.

Reduction of Nb_2O_5 by zinc to Nb_2O_3 , see Osborne, *Am. Journ. Sci.*, 1885, xxx., (3), p. 333.

One m.-gram. Nb as Nb_2O_5 , a mixture of 2 m.-grms. Nb and 100 m.-grms. Ti, and one of 1 m.-gram. Nb and 100 m.-grms. Ti were, in separate experiments, dissolved in HF , 1 c.c. H_2SO_4 (1:84) added, and the mixture evaporated till the acid fumed; the solution was then treated with HCl and Zn, and tested with HgCl_2 by P. 25; a distinct silky white precipitate formed immediately in each case.

The experiment was repeated with 100 m.-grms. Ti, 10 m.-grms. Ta (previously freed from niobium by precipitation as $2\text{K}_2\text{TaF}_7 \cdot \text{Ta}_2\text{O}_5$), and with 100 m.-grms. Sn as oxides, and with 300 m.-grms. Zr as $\text{Zr}(\text{SO}_4)_2$ in separate experiments; in every case the HgCl_2 solution remained perfectly clear. The experiment was several times repeated with 300 m.-grms. Ti as H_2TiO_3 from three different commercial sources; no precipitate formed at once or for a

few minutes in the HgCl_2 solution, but a turbidity always appeared in fifteen minutes, and this increased on longer standing.

The experiment was repeated with 100 m.-grms. Mo as $(\text{NH}_4)_2\text{MoO}_4$, with 100 m.-grms. V as Na_2VO_4 , and with 4 m.-grms. W as Na_2WO_4 in separate experiments; a large precipitate was produced in the HgCl_2 solution in each case.

For evidence of the reduction of WO_3 to WO_2 , see v. d. Pfordten, *Ber. Chem., Ges.*, 1883, xvi., p. 508; of MoO_3 to MoO_2 (when air is not completely excluded), see v. d. Pfordten, *Ber. Chem. Ges.*, 1882, xv., pp. 1925—1929; of V_2O_5 to V_2O_4 , Roscoe, *Ann. Chem. Suppl. Bd.*, 1867, vi., p. 94. The colour changes are also there described.

Mixtures of 300 m.-grms. W and 300 m.-grms. Ti, of 5 m.-grms. W and 300 m.-grms. Ti, and of 300 m.-grms. Ti and 300 m.-grms. Mo, in separate experiments, were dissolved in HF , and treated by P. 33, 35, 36, and 38; in every case a rather large precipitate was produced when the third of the solution was treated with Zn and tested with HgCl_2 . After the fusion of the remainder with Na_2CO_3 and S, the second test with Zn and HgCl_2 resulted in no immediate precipitate in the case of the last two of these mixtures, and in a slight turbidity (less than that given by 1 m.-gram. Nb) in the first mixture. The experiment with this first mixture was repeated with the same result.

Two m.-grms. Nb and 200 m.-grms. Ti in one experiment and 2 m.-grms. Nb alone in another were dissolved in HF , evaporated with 2 c.c. H_2SO_4 (1:84), and treated by the last paragraph of P. 38; a considerable precipitate of HgCl_2 was obtained in each case, that with the niobium alone being smaller, probably owing to difficulties of manipulation in the process. But in some of the test analyses (see the next chapter), 2 m.-grms. Nb disappeared between the first and second tests with HgCl_2 in the processes connected with the fusion.

P. 38, N. 5.—A mixture of 300 m.-grms. Ti as H_2TiO_3 , and 5 m.-grms. W as Na_2WO_4 in HF solution, was treated by P. 33 and 35. The $(\text{NH}_4)_2\text{S}$ filtrate was then acidified with H_2SO_4 (1:20), the precipitate tested for tungsten by P. 43, and the filtrate by P. 40; no precipitate (of H_2WO_4) was obtained in either case. The $(\text{NH}_4)_2\text{S}$ precipitate was then fused with Na_2CO_3 and S, and the fused mass treated with water as in P. 38; the aqueous solution was acidified with H_2SO_4 (1:20), and the precipitate tested by P. 43 and the filtrate by P. 40; no precipitate of H_2WO_4 resulted from the treatment of H_2SO_4 precipitate, but a small, though very distinct, compact yellow residue was obtained from the filtrate. The residue from the Na_2CO_3 and S fusion that was insoluble in water was dissolved in HF by a half-hours' digestion, and treated by the first paragraph of P. 38; no precipitate of HgCl_2 was obtained, showing that the tungsten had been completely separated from the titanium by the Na_2CO_3 and S fusion.

For the complete separation of tin and tungsten from niobium and tantalum by fusion with Na_2CO_3 and S, see Hall, "Observations on the Metallic Acids," 1904, p. 9, thesis submitted to the University of Pennsylvania.

(To be continued.)

Method of Detecting very Small Quantities of White Phosphorus.—Rudolf Schenck and E. Scharff.—White phosphorus, even when present in very small quantities, ionises the air, while P_4S_3 , which resembles it in luminescence, has no such action. This property can be employed to detect very small quantities of white phosphorus in P_4S_3 . The presence of 0.004 m.-gram. can be demonstrated by this test, and it is thus far the most delicate known. By slowly burning the sesquisulphide no P_4O_6 is formed, and this is the cause of the difference in the actions of P_4S_3 and white phosphorus.—*Berichte*, xxxix., No. 6.

A REVISION OF THE ATOMIC WEIGHTS OF SODIUM AND CHLORINE.*

By THEODORE WILLIAM RICHARDS

and
ROGER CLARK WELLS.

(Continued from p. 239).

THE RATIO OF SODIC CHLORIDE TO SILVER.

SINCE the time of Gay-Lussac, the method of titration has been a favourite way of determining ratios between silver and soluble halogen salts. If the end-point were definite, this method would be capable of extreme accuracy. It involves no transference, collection, or weighing of a precipitate; consequently the introduction of silica or alkali from a glass-stoppered flask, occlusion of sodic nitrate, loss of asbestos, and other sources of error, are altogether avoided. After weighing the factors in the proper proportions, dissolving and precipitating, one has only to withdraw a portion of the mother-liquor and determine whether or not the true end-point of the reaction has been reached. It is not strange, therefore, that many eminent experimenters adopted this method in order to determine many atomic weights. The solubility of silver chloride, however, was a fact which Gay-Lussac, Pelouze, Dumas, and many others, as well as Stas in his earlier experiments, did not consider; in consequence, all used the wrong end-point. In his last experiments Stas, recognising the solubility of argentic chloride, attained the end-point in two ways. The first method consisted in titrating to the extreme limit with sodic chloride, and then running back to the opposite extreme limit with silver nitrate; the true end-point was assumed to be half-way between the two limits.† The second method consisted in bringing the mother-liquor to such a point that two portions of it gave equal opalescent precipitates with silver nitrate and sodic chloride respectively; this was the end-point established by Mulder. Before accepting this latter method, Stas attempted to prove for himself that a solution of silver chloride in water gave an equally intense opalescent effect with an excess of argentic nitrate and sodic chloride. He concluded that the opalescence in the two cases was identical, but at the same time admitted that the opalescent precipitates were extremely unstable, and varied continuously in appearance from the moment of precipitation to the time they had settled out completely ("Oeuvres," i., 158). At first, not hoping to improve essentially in this respect upon the methods of Stas, we used this end-point, and after a few preliminary trials made eight careful determinations, using in all 33.1035 grms. of pure salt and 61.0898 grms. of pure silver. The atomic weight of sodium calculated for the average of these experiments was 23.032, with maximum and minimum of 23.040 and 23.023 respectively.

These experiments were of value as giving practice in the manipulation, but the extreme variation was so much greater than it ought to have been that clearly some undiscovered cause of variable error was present in the results.

A study of all the possible causes of irregularity eliminated all probabilities except the uncertainty of the end-point. The purity of the salt and the silver left nothing to be desired, for the same samples gave constant results when the argentic chloride was weighed. Occlusion had been precluded by the use of solutions of not much over decinormal strength, and the mechanical operations were so extremely simple as to leave little opportunity for error. Hence much time was spent in investigating the opalescent phenomena attending the precipitation of traces of argentic chloride. The improved and precise nephelometer served excellently in this tiresome research (see Richards and Wells, *Am. Chem. Journ.*, 1904, xxxi., 235). We found that its readings could always be depended upon, and that if any irregularity occurred the fault was with the precipitate within the liquid, and not in the means of observing this precipitate.

The first step in these tests was to prepare a solution of

pure argentic chloride, free from excess either of silver or of chlorine. For this purpose pure floccy silver chloride was very thoroughly washed in a flask by decantation. From time to time two nephelometer tubes were filled with the clear decanted wash-water; to one was added a millilitre of argentic nitrate solution, which contained a milligram of silver, and to the other a millilitre of an equivalent solution of sodic chloride. After stirring, these tubes were examined in the nephelometer within ten or fifteen minutes, and the intensities of the two opalescences were compared. No regularity could be detected in the results, successive trials varying over 50 per cent from one another. For example, in one trial the tube containing an excess of silver was twice as intense in opalescence as the other; while in the following trial, with equal proportions of the same solution, it was only two-thirds as intense as the other. The average of a dozen comparisons confirmed approximately Stas's and Mulder's statement that argentic chloride gives equal opalescence with the two precipitates, but the wide variations between the extremes emphasizes Stas's conclusions as to the uncertainty of the results.

Subsequent experiments showed that the presence of an electrolyte greatly accelerated the formation of the precipitate, indicating a colloidal transition stage. The preceding experiments had been almost free from electrolyte, and in view of this inference, it is easy to see why the formation of the precipitate had been so irregular, for slight differences in the extent and speed of mixing might cause important differences in the coagulation of the colloid. Clearly, in making nephelometric experiments, an excess of electrolyte must be present. Other possible causes of error, such as the effect of light and the presence of absorbed argentic nitrate or common salt in the precipitated argentic chloride from which the solution was made, were carefully guarded against in the further experiments. In order to avoid possible hydrolysis, nitric acid was always added.

(Mulder, "Die Silber-Probirmethode," 1859, p. 72. Some of Mulder's observations in this direction may have been due to occluded material, but the danger of hydrolysis is not wholly to be despised).

Even when all these precautions had been taken, however, incomprehensible irregularities were still present in the results. It was then found that the time allowed for the formation of the opalescence had not been adequate; that in some cases one tube had clouded much faster than the other, but that in all cases neither tube had attained maximum intensity. Often as much time as eight hours was needed for the attainment of this maximum; but the time must not be too greatly extended, or some of the precipitate will be deposited on the bottom of the tube. The performance of the precipitation when chloride is in excess is thus not wholly parallel with that when silver is in excess, already discussed on p. 202.

In view of these facts a new comparison was made. A quantity of silver chloride was precipitated in red light from cold decinormal solutions of silver nitrate and sodic chloride, washed twelve times with water weakly acidified with nitric acid, and eight times further with pure water, the last washings being made in absolute darkness. Pure water containing such quantities of nitric acid and sodic nitrate as would occur in an actual mother-liquor from a quantitative precipitation, was then added, and the mixture was allowed to stand with occasional agitations for a day. In making the nephelometer observations on the decanted liquid, sufficient time was allowed for the precipitates to become constant. Three completely separate trials were made in the nephelometer, and of these are recorded below the depths of liquids which seemed to be equally opalescent.

Observation.	Time.	Tube + AgNO ₃ .	Tube + NaCl.
65	After eight hours	98 m.m.	100 m.m.
66	" "	100	100
67	" "	103	100
Average		100.3	100

* Published by the Carnegie Institution of Washington, April, 1905.
† "Oeuvr.," i., 755. This is unsatisfactory for many reasons. See Richards, *Proc. Amer. Acad.*, 1893, xxix., 82.

These observations, made with great care, settled every doubt about the correctness of the equal opalescence end-point. Thus it is clear that Stas encountered difficulties with this method only because he, like all the previous experimenters, did not wait long enough to allow the opalescence really to establish itself in the absence of electrolyte to hasten precipitation.

In the course of these experiments several subordinate points were noted which are worth recording. When the opalescent precipitates were made directly in the tubes from sodic chloride and argentic nitrate, having first one and then the other in excess, the speed of formation was not like that observed when a solution of argentic chloride was treated with the two electrolytes. In the former case, the maximum opalescence was attained more slowly, and the tube containing an excess of chloride always became cloudy much more rapidly than that containing an excess of silver, while in the latter case the difference was much less marked. This adds to the evidence that the dissolved argentic chloride is appreciably colloidal, and that the formation of this colloidal portion requires time.

The possible effect of light upon this end-point was another circumstance of interest, although not of immediate bearing on our quantitative work, since actinic light had been scrupulously excluded. In order to ascertain the effect of light, the solution already tested in Observations 65, 66, and 67 was agitated in the flask for an hour at a distance of about 30 c.m. from a white incandescent electric light. Observations 68 and 69 were made with the mother-liquor. It was then agitated in diffused daylight for another hour, an exposure which made the precipitate decidedly bluish, and Observations 70 and 71 taken.

Observation.	Tube + AgNO ₃ .	Tube + NaCl.
68	100 m.m.	100 m.m.
69	100	100
70	97	100
71	100	100
Average ..	99	100

The solution thus gave the same result in the nephelometer as before. Böttger, in his study of the solubility by the conductivity method, also observed that the conductivity did not sensibly alter until the conductivity vessel was exposed to strong light for a considerable length of time. A slight colouring of the precipitate made no difference (*Zeit. Phys. Chem.*, 1904, xlv., 521). The explanation must be that chlorine thus set free does not ionise. According to Vogel (*Gilbert's Annalen*, lxxii., 286), silver sub-chloride is insoluble in very dilute nitric acid. Hence neither an excess of ionised silver nor chloride should appear in the mother-liquor. Light, therefore, could not have been the cause of any of the variations in preceding observations, and its complete exclusion had been supererogatory.

Having thus, by many experiments, of which those given above are merely samples, assured ourselves that the true end-point may be decided by equality of opalescence when sufficient time is allowed to complete the opalescent precipitation, it became an important matter to make a conclusive series of analyses by this method.

In the first place, a given piece of the purest silver was carefully weighed, and was dissolved in a flask upon the steam-bath. For every grm. of silver about 3 millilitres of nitric acid, of density about 1.20, were used. This density was secured by mixing pure distilled nitric acid of the usual concentration with an equal volume of water. After the silver was dissolved, more water was added, and the nitrous fumes were expelled by a brief very gentle ebullition. A tower of bulbs, ground to fit the neck of the flask, prevented any loss of silver nitrate during the operations. A special experiment proved the efficiency of the bulbs as a means of retaining spray. In this manner of treatment nitric acid always remained in slight excess.

Assuming a probable value for the atomic weight of sodium, the exact weight in air of salt equivalent to the weight of the given piece of silver was calculated and weighed out for fusion. There was always a slight loss of weight when the salt was fused in the platinum crucible, and if, after fusion, more than a m.grm. of salt was still required, approximately the required amount of well-dried salt was added at once by a platinum spatula. The amount so added never exceeded a few m.grms., and its introduction could have caused no error, since in our laboratory salt was not hygroscopic. The exact weight of salt corrected to vacuum was obtained. Any further slight lack of equivalence was then made up by volumetric addition of the proper quantity of the same solutions which were to bring the mother-liquor to equal opalescences after the precipitation. Of course the recorded weights include all these small additions.

Both salt and argentic nitrate were diluted to a concentration about fifth normal, and were then mixed in complete darkness with all possible precautions, the argentic nitrate being very slowly poured into the salt solution. When in a day or two the precipitate had settled and the mother-liquor had become clear, the latter was examined nephelometrically.

It was found to be convenient, in determining the amount of a deficiency of either silver or chloride, to calculate once for all the extent of this deficiency in terms of the ratio of lengths of the two nephelometer columns giving equal apparent opalescence. This was in the first place calculated by means of the law of concentration effect; but practically a slightly different value, based upon experience, was found to be more accurate. A ratio of 100 m.m. to 80 m.m. was found to signify an excess or deficiency of 0.15 m.grm. of silver per litre, and other amounts in proportion. Since the average of many readings was probably accurate within 2 per cent and the volume rarely exceeded a litre, it would appear that the end-point was determined within 0.02 m.grm. of silver. This does not, of course, prove that the results are accurate to within this small margin, because other experimental details may have led to larger error. The difference between the maximum and minimum results corresponds, indeed, to a much greater uncertainty, as will be seen. Hence the end-point is determined precisely enough for our purpose.

(To be continued).

PROCEEDINGS OF SOCIETIES.

SOCIETY OF CHEMICAL INDUSTRY. (LONDON SECTION).

Ordinary Meeting, May 7th, 1906.

Mr. A. GORDON SALAMON in the Chair.

"Notes on the Gutzeit Test for Arsenic." By J. A. GOODE and F. MOLLWO PERKIN, Ph.D.

Owing to the difficulty of obtaining zinc free from arsenic the authors used an ammonium salt—preferably the chloride—and metallic magnesium. Numbers are given showing the solution potential of magnesium in ammonium chloride, and sulphuric and hydrochloric acids, also the difference produced by the addition of cadmium salts. The potential found was always lowered by the addition of the cadmium salt.

Attempts to obtain a permanent stain were unsuccessful. The authors, however, do not consider that this is a matter of great importance, because as the test is so readily carried out it is easy to conduct several experiments simultaneously. One to produce a standard stain, one a blank, and the other with the substance under examination.

The authors found that mercuric bromide is more

delicate than mercuric chloride and the stain is more intense. With mercuric bromide it is possible to detect 1/2000 m.grm. of arsenic.

Although magnesium and ammonium chloride were employed by the authors, they also used zinc and acids, and obtained results equally as good. In fact, they consider that zinc and acid is fractionally more sensitive than magnesium and an ammonium salt.

"The Separation of Brucine and Strychnine by Nitric Acid—Influence of Nitrous Acid." By W. C. REYNOLDS, B.Sc., A.R.C.S., and R. SUTCLIFFE.

The authors have examined the processes proposed by Keller (*Zeit. Oesterr. Apoth. Ver.*, 1893, 542), Stoeder (*Ned. Tydschr. Pharm.*, 1899, xi., 1—5), and Gordon (*Arch. Pharm.*, 1902, ccxi., 641), and show under what conditions brucine can be completely oxidised with the minimum loss of strychnine. They have also investigated the part played by nitrous acid in the oxidation, and the action of alkalis on the products.

"Absorption of Gallic Acid by Organic Colloids." By W. P. DREAPER, F.I.C., and A. WILSON, B.Sc.

The absorption of gallic acid by silk and hide powder is shown to be of a similar nature to its absorption by gelatin or albumin. The influence of general reagents and the curves obtained indicate that the reactions are due to absorption. The precipitation of these colloids by tannic and gallic acids indicates when studied in detail that the solution state is a determining factor in the production of these coagula. The influence of gallic acid on the nature of a tannic acid gelatin coagulum is also observed. The results confirm the pseudo solution theory of dyeing, and indicate the nature of tanning.

FARADAY SOCIETY.

Ordinary Meeting, May 15th, 1906.

Dr. F. MOLLWO PERKIN, Treasurer, in the Chair.

Mr. H. D. LAW, B.Sc., read a paper entitled, "*Behaviour of Platinised Electrodes.*"

The author desired to find an electrode on which the reduction of the aromatic aldehydes and similar easily reducible compounds could not be effected. Platinised platinum, as being the metal from which hydrogen is liberated at the lowest potential, was tried as the cathode in an acidified alcoholic solution of benzaldehyde. At first, energetic reduction took place; the activity of this, however, diminished in successive experiments, and was extremely small after twelve hours' polarisation. The cause of the reaction is obscure; it is probably catalytic in its origin.

Dr. N. T. M. WILSMORE drew attention to Tafel's work, which the author's results appeared to contradict.

Dr. F. MOLLWO PERKIN remarked on the differences between lead and platinum electrodes, and discussed the general bearing of the author's experiments.

Mr. JULIUS L. F. VOGEL, M.I.E.E., M.I.M.M., read a paper on "*The Electrolysis of Fused Zinc Chloride in Cells Heated Externally.*"

The author begins by explaining that the work which formed the subject of the paper was carried out for the most part between 1898 and 1901, and could be conveniently divided into three periods. He then explains how the investigation came to be undertaken; the original inception of the process was due to the late F. Maxwell Lyte, who took out patents for a complex ore process involving the electrolysis of fused zinc chloride and for the dehydration of zinc chloride. The process as such was never investigated practically. The second period deals with the investigation of the dehydration of zinc chloride by evaporating under reduced pressure, and the electrolysis of the salt in a fused state in externally heated cells by Dr. O. J. Steinhart and the author jointly on behalf of the Smelting Corpora-

tion, Ltd. The various experiments and the apparatus employed for these experiments are described. The third period deals with the further investigations made after the United Alkali Company had joined the Smelting Corporation in testing the process, and details are given of the work as carried out under the joint supervision of the author's firm and the chemical staff of the United Alkali Company. The author describes how the process was carried successfully to a stage when continuous electrolysis was carried on for eleven days and nights, and three hundredweight of pure zinc was produced.

He then explains how, on the failure of the Smelting Corporation, the work was suspended, and finally abandoned, although further elaborate investigations were undertaken by the United Alkali Company utilising cells heated internally by the current.

The author finally reviews the work done in cells heated externally, and expresses his opinion that the process was "non-proven," and that it still offers possibilities of being carried to commercial success.

The paper is illustrated with sectional drawings of apparatus, and lantern slides, prepared from photographs of the actual plant, were exhibited.

Dr. O. J. STEINHART referred to the production of zinc from zinc oxide by feeding the latter into a molten bath of chloride, after the fashion of aluminium electrolysis.

Mr. W. MURRAY MORRISON mentioned some of the advantages of internal heating with regard to local inequalities of temperature.

Dr. H. BORNs referred to the formation of zinc clouds diffused in the electrolyte under certain conditions, and consequent reduction of efficiency, observed by Lorenz. He asked whether the addition of impurities, such as ammonium chloride, had been tried. Evaporating with HCl was a method of dehydrating the chloride.

Mr. L. GASTER asked for information regarding comparative energy losses in externally and internally heated cells, and also regarding the consumption of carbon.

Dr. G. RUDORF (communicated), after reviewing the various methods proposed for dehydrating the chloride, affirmed that the author's was the only practicable one. He asked for information regarding the effect on the enamel of continuous heating with zinc chloride; his experience was that a perfect enamel was not obtainable.

Mr. J. L. F. VOGEL, in reply, said that the use of a fused metallic cathode and proper temperature and other conditions prevented the formation of zinc cloud. Carbon losses depended greatly on the dehydration; they had not had opportunities of trying Acheson graphite. It was undesirable to add foreign substances, as these would accumulate in time; it was likewise impracticable to dehydrate in the fashion referred to by Dr. Borns. The difficulty in electrolysing a solution of ZnO in fused chloride was that the solubility was slight, and at the high-current densities demanded by practice not sufficient oxide would pass into solution.

NOTICES OF BOOKS.

The Principles and Practice of Iron and Steel Manufacture. By WALTER MACFARLANE, F.I.C. London, New York, and Bombay: Longmans, Green, and Co. 1906.

The scope of this book coincides very nearly with that of the Iron and Steel Manufacture Syllabus of the City and Guilds of London Institute, and it will be found of great use to the student who is taking such a course of instruction. The practical aspect of the subject receives special attention, the author's experience in iron and steel works having enabled him to go into many of the processes more minutely than is usual in books of this kind, and at the same time to make his accounts both vivid and

interesting. A novel feature characterising the book is the reversal of the usual order of treating the subjects, so that the working and selection of pig-irons is considered in the first part, while the second part deals with the composition of the ores, the construction and equipment of the blast-furnace, and the production of pig-iron; this is a plan which possibly has some advantages to recommend it, and in any case the order of the two sections could readily be inverted, as each section, and indeed each chapter, is quite complete in itself. A special chapter by H. W. Waldron on the treatment of tool-steel gives a short but interesting account of the hardening of steel in practice. The chief defect of the book appears to be the devotion of much space to the most elementary chemical considerations with which every student nowadays would probably be perfectly familiar before he began his technical training, and even supposing he were quite ignorant of chemistry, he would not find enough in the book to explain the reactions occurring during the manufacture of iron, and would consequently be bound to fall back upon the ordinary text-books for further information.

Metallurgical Calculations. By JOSEPH W. RICHARDS, A.C., Ph.D. Part I. New York: McGraw Publishing Company. 1906.

THE series of articles which appeared from March, 1905, to March, 1906, in the *Electrochemical and Metallurgical Industry*, and of which the greater part of this book is a reprint, was well worth preserving in the more convenient form in which it is now published. The importance for metallurgists of acquiring skill in making calculations cannot be over-estimated, not only for the purpose of determining the real efficiency of the various processes, for which it goes without saying that such calculations are absolutely essential, but also for the consolidation of their theoretical knowledge. For the latter purpose, the collection of unworked problems contained in this book will be found well adapted, many of the examples being of considerable difficulty. The book is also worthy of the attention of others besides metallurgical chemists, for it contains many useful hints on the most expeditious methods of solving the mathematical problems met with in general chemistry; thus, examples are included on weights and volumes of gases, and on elementary thermo-chemistry, for which tables of heats of formation are given. The applications of thermo-chemistry occupy a large part of the book, and artificial furnace-gas is also treated very fully, while the principles of the conduction and radiation of heat are not neglected.

Grundriss einer Entwicklungsgeschichte der Chemischen Atomistik. ("Outlines of the History of the Development of the Chemical Atomistic Doctrine"). By Dr. RICHARD EHRENFELD. Heidelberg: Carl Winter. 1906.

THIS introduction to the study of the history of chemistry is marked by great thoroughness, and will appeal more especially to those who are interested in the investigations and theories of the earliest natural philosophers. Beginning with the speculations of Anaxagoras and Empedocles, the author traces the gradual development of the atomistic view of the ultimate constitution of matter as expounded by the three Grecian atomistic philosophers, and gives a review of Plato's Theory of Matter and Aristotle's Doctrine of the Elements. Passing to later times, we find Boyle's Corpuscular Theory fully worked out in connection with the systems of Descartes and Gassendi; the work of this last philosopher, whose claims to recognition are often overlooked, is discussed fully, and a just estimate of its true value given. The treatment of Dalton's hypothesis presents no specially new features, while the chapters on the electron theory are very slight, although the author shows that, had he allowed himself more space for the discussion of this branch of the subject, he might have produced an interesting account of the work of modern investigators in this region.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Berichte der Deutschen Chemischen Gesellschaft.
Vol. xxxix., No. 6, 1906.

Properties of Liquid Nitrogen.—H. Erdmann.—Liquid nitrogen is a very mobile clear colourless liquid, differing markedly in its physical properties from liquid air; this latter contains much oxygen, and its specific gravity is so high that a piece of ice thrown into it always floats, while in liquid nitrogen both ice and absolute alcohol sink as soon as the temperature has adjusted itself, and the violent evolution of gas, which first of all forces the frozen alcohol and ice to the surface, has ceased. This phenomenon is in better agreement with the specific gravity given by Ramsay and Drumman (0.7914 at the boiling-point) than with that determined by Dewar (0.850). If liquid air is poured over a bulb filled with dry oxygen at ordinary pressures and then closed, no visible change occurs in the interior, but if liquid nitrogen is used the oxygen at once condenses into bluish drops. Thus the temperature produced by the evaporation of liquid nitrogen is lower than that produced by liquid air, and for this reason it is a very good cooling agent. It is a good solvent for liquids boiling at a low temperature. The miscibility of liquid nitrogen with liquid ozone is very striking. A 15 to 20 per cent solution of ozone in liquid oxygen remains perfectly clear after the addition of many times its volume of liquid nitrogen, but if ozonised oxygen is led directly into liquid air, phenomena occur from which it might appear that ozone is salted out from its solution in oxygen by means of liquid nitrogen. Solutions of ozone in liquid nitrogen might be used to determine the molecular weight of ozone in solutions. The author has determined by the surface tension method the molecular weight of liquid nitrogen, but will not publish the results until he has repeated the experiments with chemically pure nitrogen of constant melting-point. Liquid nitrogen appears to be as indifferent as the gaseous element, extinguishing a burning chip and magnesium wire, but if poured over burning metallic calcium, calcium nitride is formed; this yields ammonia with water.

Action of Hydrogen Peroxide on Phosphorus.—Th. Weyl.—Yellow phosphorus when warmed with water yields hydrogen phosphide. Six per cent hydrogen peroxide begins to act on yellow phosphorus at 60°, the resulting products being hydrogen phosphide, which is not spontaneously inflammable, phosphorous acid, and phosphoric acid. The excess of phosphorus which has not taken part in the reaction is converted finally into a horny mass, which yields hardly any fumes in the air, but burns in a flame like ordinary phosphorus. The action of hydrogen peroxide on amorphous and on Schenck's phosphorus is far more energetic. When a peroxide containing more than 8 per cent is used, the action is very violent, and much heat is evolved. The products of the action are hydrogen phosphide, phosphorous acid, and phosphoric acid. When boiling water acts on amorphous phosphorus, hydrogen phosphide is formed, and also (and in much greater quantity) when 5 per cent caustic soda acts on amorphous phosphorus. This development of hydrogen phosphide from amorphous and Schenck's phosphorus by the action of boiling water, caustic soda, and hydrogen peroxide is not to be ascribed to the presence of yellow phosphorus in the preparations, for equal amounts were obtained when all traces of yellow phosphorus were previously removed. Possibly phosphorous acid is first formed according to the equation $3\text{H}_2\text{O}_2 + 2\text{P} = 2\text{P}(\text{OH})_3$, and this is then converted into hydrogen phosphide and phosphoric acid as follows:— $4\text{P}(\text{OH})_3 = \text{PH}_3 + 3\text{PO}(\text{OH})_3$, while a part of the phosphorous acid is oxidised to phosphoric acid by the excess of hydrogen peroxide.

Constitution of Chromic Acid.—W. Manchot.—On investigating quantitatively the oxidation of uranous oxide by means of chromic acid in presence of hydriodic acid it is found that one atom of uranium replaces three equivalents of oxygen, using up two equivalents itself, while the other is transferred to the hydriodic acid. The three equivalents are released simultaneously, which leads to the conclusion that in chromic acid the three equivalents of oxygen are directly connected with one another. Hence the formulæ appear to be $O=Cr<\overset{O}{\underset{O}{\parallel}}$ and $HO>Cr<\overset{O}{\underset{O}{\parallel}}$

rather than $Cr\overset{O}{\parallel}=\overset{O}{\parallel}O$.

Oxidation of Ammonia to Compounds of Nitrogen and Oxygen.—Otto Schmidt and Rudolf Böcker.—The authors have investigated the oxidation of ammonia to nitric acid, nitrous acid, and hyponitrous acid by the use of catalytic agents, paying special attention to the yield. Using platinum and platinum asbestos they obtained 75 to 76 per cent of the ammonia used as oxygen compounds, and in some cases more than 80 per cent. The first product is nitric oxide. This is then rapidly converted by the excess of oxygen into nitrous acid anhydride, which amounts to about 80 to 90 per cent of the total products, the residue being nitric acid. Bayer has found that ammonia is oxidised by the air or by oxygen in presence of pyrites under certain conditions, and it seems probable that this might be a profitable process to employ commercially, as the yields are good. On the other hand, Ostwald's process of oxidising ammonia by free oxygen to nitric acid by contact substances, e.g. platinum, would not be a lucrative process at present prices.

Atomic Weight of Nitrogen.—Philippe A. Guye.—Determined by physico-chemical methods the mean of many experiments gave for the atomic weight 14.008, and by chemical methods it was found to be 14.010. The International Atomic Weight Committee has retained the number 14.04, which the author believes to be erroneous for the following reasons:—1. It was determined by the old methods, which rested on very indirect relations between the atomic weight of nitrogen and that of silver. These methods were not exact enough to guarantee the accuracy of the second figure in the decimal. 2. The new value $N = 14.01$ results from twelve determinations of the direct ratio to oxygen, and this element is the actual basis of all other atomic weights. Six of these determinations were performed by physico-chemical and six by chemical methods. 3. Chemists have always been in the habit of taking $N = 14$ for rough calculations, and this differs from the true value by only 1/1400, and, moreover, agrees perfectly with the accurately determined densities of the gases nitrogen, nitrous oxide, nitric oxide, and ammonia, and with exact analyses of the gases nitrous oxide and nitric oxide.

Constituent of Phosphorised Air which causes Electric Conductivity.—Rudolf Schenck, F. Mihr, and H. Banthien.—The examination from the chemical point of view of the substance which causes the ionisation of the air in the neighbourhood of white phosphorus has shown that it cannot be due to ozone. Nor does the oxidation of organic substances, such as oil of turpentine, cotton, wood, &c., by ozone produce any conductivity, although it is accompanied by luminescence phenomena. The authors have examined a whole series of oxidation processes which closely resemble those of phosphorus; e.g., that of bromoacetylene, CH_2Br , and have found no trace of the formation of ions. Thus, apparently the oxidation process in the case of white phosphorus is not itself the cause of the ionisation, which is to be sought rather in the secondary processes which are possibly conditioned by the occurrence of oxidation processes. It has been found that the conductivity continues only as long as oxidation is going on. All factors which prevent oxidation also prevent the appearance of the conductivity phenomenon; e.g., oil of turpentine, carbon disulphide vapour. It is

known that the oxidation of white phosphorus ceases in pure oxygen at atmospheric pressure. The authors have now shown that pure oxygen which has passed over phosphorus causes a very much smaller leakage of electricity than air under the same conditions. The addition of small quantities of ozone to the pure oxygen causes a powerful increase in the conductivity, and the same conductivity is obtained if pure air, after it has been in contact with white phosphorus, is led over or through the following substances:—Oil of turpentine, alcohol, mesitylene, ammonia, ethylene, amylene, &c. The cause of the conductivity thus lies, not in the oxidation process as such, but in the presence of an oxidation product of phosphorus. The substances which prevent oxidation prevent the formation of the product, but cannot in themselves influence the ionisation. Red phosphorus also yields the so-called phosphorus emanation. The authors have investigated the action of the vapours of phosphorus trioxide on the electroscope, and have found that the action is very strongly marked, so that there can be no doubt that this substance is responsible for the conductivity.

MISCELLANEOUS.

Estimation of Cadmium in Volatile or Organic Salts.—H. Baubigny.—When a cadmium salt precipitated from its solution by hydrogen sulphide gas is a volatile one, such as the chloride or bromide, a distinct error in estimation may be caused, owing to traces being left in the sulphide. In order to eliminate this error it is necessary to obtain the sulphate from the sulphide by a very careful method. The sulphide is detached from the filter after washing and without previous desiccation. The precipitate rapidly sinks in water, and the clear liquid above can be decanted. After thorough washing by this method the merest trace remains on the filter-paper, and therefore a negligible trace of volatile salt. After incineration of the paper, the residue can be added to the sulphide and the whole be transformed into sulphate. By this method the evaporation of the washing water is avoided which would necessitate the re-dissolving of the sulphide on the filter. The results obtained are most accurate. In the case of an organic salt of cadmium a decided excess of sulphuric acid is added to the solution. If this acid is insoluble, it is filtered and washed with acidulated water; if it is soluble, as the decomposition of the salt is almost integral in presence of sulphuric acid, the method may be proceeded with as in the former case without any fear of loss.—*Comptes Rendus*, cxlii., No. 17.

MEETINGS FOR THE WEEK.

- TUESDAY, 5th.—Royal Institution, 5. "Northern Winter Sports—Sweden and its People," by Col. V. Balck.
WEDNESDAY, 6th.—Society of Public Analysts, 8.
THURSDAY, 7th.—Royal Institution, 5. "Man and the Glacial Period," by Prof. W. J. Sollas, F.R.S.
— Chemical, 8.30. "Ammonium Selenate and the Question of Isodimorphism in the Alkali Series," by A. E. H. Tutton. "An Improved Beckman Apparatus for Molecular Weight Determination," by J. M. Sanders. "Resolution of Lactic Acid by Morphine," by J. C. Irvine. "Vapour Pressures of Binary Mixtures—Part I., Possible Types of Vapour Pressure Curves," by A. Marshall. "Action of Sodium on α -Dichloropropylene," by I. Smedley. "Thiocarbamide as a Solvent for Gold," by J. Moir. "Action of Sulphur Dioxide and Aluminium Chloride on Aromatic Compounds," by S. Smiles and R. Le Rossignol.
FRIDAY, 8th.—Royal Institution, 8. "Studies on Charcoal and Liquid Air," by Prof. Sir James Dewar, D.Sc. F.R.S., &c.
— Physical, 8. "Solution of Problems in Diffraction by the Aid of Contour Integration," by H. Davies. "Effect of Radium in Facilitating the Visible Electric Discharge in vacuo," by A. A. Campbell Swinton. Mr. J. Gould's Experiments with a Vibrating Steel Plate, exhibited by Messrs. Newton and Co. "Fluid (Liquid) Resistance," by Col. de Villamil.
SATURDAY, 9th.—Royal Institution, 3. "Inspiration in Poetry," by Prof. W. Macnellan Dixon, M.A., &c.

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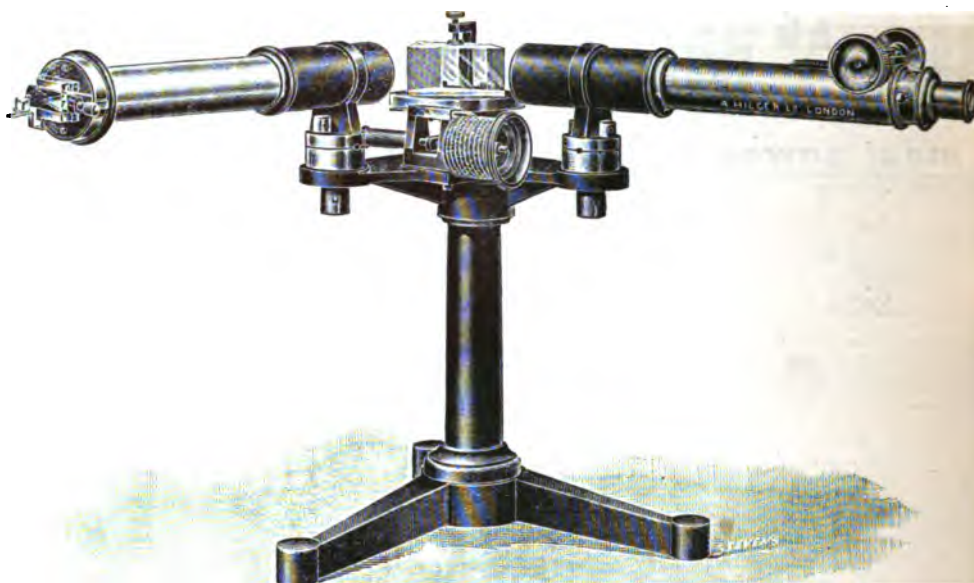
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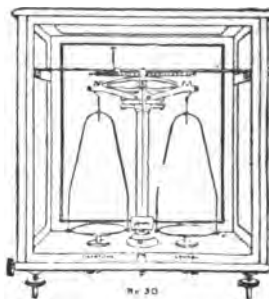
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THE CHEMICAL NEWS.

VOL. XCIII., No. 2428.

A NEW PRODUCT OF ACTINIUM.

By O. HAHN.

RECENTLY the attention of investigators has been drawn to the remarkable similarity in the nature of the radio-active decay of thorium and actinium.

Thorium, itself inactive, yields as its first product radio-thorium (O. Hahn, "Jahrb. Radioact. Elektr.," II., 330), which emits α -rays. Then follow thorium X, the characteristic emanation, and finally the "active deposit." (for this expression see Hahn, "Ueber Einige Eigenschaften der α -Strahlen des Radiothorium," *Physikal. Zeit.*). Actinium behaves in exactly the same way. By means of the same methods as led to the separation of thorium X from thorium, Godlewski (*Phil. Mag.*, July, 1905), and, independently, Giesel (*Berichte*, 1905, xxxviii., 775) separated from actinium a new product, actinium X. This yields the emanation, which in its turn gives the active deposit.

Moreover, the similarity between actinium and thorium is still greater. I have succeeded in finding in actinium a new product which lies between actinium and actinium X, and which may be called "radio-actinium" from analogy with thorium. The substance emits α -rays, decays to the half value in about twenty days, and then yields actinium X, which in its turn decays to the half value in 10.2 days.

The separation of radio-actinium from an actinium solution in radio-active equilibrium is effected as follows:—A very fine precipitate is produced in the solution, and this gradually settles and carries down the new product with it, while the greater part of the actinium and actinium X remains in solution. Amorphous sulphur was found very suitable for this purpose. A small quantity of sodium thiosulphate was added to a fairly strong acid solution of actinium and hydrochloric acid, and the slight precipitate was allowed to settle in the cold. After filtering and washing, the precipitate was examined for activity. It showed a strong α -radiation, but, comparatively speaking, hardly any β -radiation nor any emanation. The activity of the α -rays increase, until after about three weeks a maximum is reached. The activity is then about two to three times as strong as directly after preparation.

The activity then begins to decrease slowly, finally with a half-value period of about twenty days, following an exponential law.

The activity of the β -rays and the power of emanation, both insignificant shortly after the preparation of the radio-actinium, increase very strongly, reach their maximum at the same time as the α -rays, and then decrease at the same rate as the latter.

The first rise of the activity to a maximum depends on the formation of actinium from radio-actinium. Thus if we separate off the actinium X when the activity of the radio-actinium begins to exhibit a decrease, the residue first becomes stronger again, and behaves in exactly the same way as the freshly prepared radio-actinium. The actinium X separated decays with its characteristic period of ten days.

Some experiments of Dr. Levin, performed in this Institute and soon to be published, are important in considering this question. He prepared actinium X in the usual way with ammonia. But he did not succeed in reducing the α -activity of the filter to the small fraction which had been obtained by others. An investigation of his still strongly active precipitate showed that its powers of emanation was practically zero, and thus actinium X was out of the question. As actinium itself has been

found to be almost inactive, the observed α -activity indicated another product, which is radio-actinium.

Actinium itself, free from radio-actinium and actinium X, emits practically no α - nor β -rays, then slowly increases in activity and reaches a maximum after about four months.

Godlewski (*Phil. Mag.*, July, 1905) obtained an almost inactive actinium, a proof that, without knowing it, he had separated the radio-actinium from it.

I have observed that on dissolving actinium in hydrochloric acid, mostly only a small quantity remains undissolved, and this small amount contains a large proportion of radio-actinium.

Giesel has recently announced that his emanum preparations increase in activity for about six months. This may possibly be explained by assuming that radio-actinium is formed.

In a communication which has recently appeared, Marckwald (*Berichte*, 1905, xxxviii., 2264) compared the chemical properties of actinium and emanum with one another, and came to the conclusion that actinium and emanum are not identical, but are genetically related, viz., actinium is a decay product of emanum, as the activity of actinium disappears in the course of a few months, but actinium and not emanum is the cause of the interesting emanation. This is contradictory to Debiere's statement that his actinium does not decrease in activity.

The decaying substance described by Marckwald, which he takes for Debiere's actinium, is possibly the new product radio-actinium, for a thorium precipitate with sodium thiosulphate brings down radio-actinium too. It is then not explained why his product did not increase in the beginning, or why he did not succeed in getting actinium X from it.

It must be mentioned that the above experiments were performed both with Debiere's actinium and also with Giesel's emanum. The result was always the same.

A full description of the experiments will be given elsewhere.—*Berichte*, 1906, xxxix., 1605.

ON THE USE OF GYPSUM FOR THE RECOVERY OF AMMONIA AS A BY-PRODUCT IN COKE MAKING.

By H. WARTH.

As there must be many localities in which sulphuric acid is not available in sufficient quantity for the production of ammonium sulphate, the following experiments were made to demonstrate the possibility of using gypsum instead of sulphuric acid. Gas-water (of 1.02 specific gravity) from the municipal gas works of Birmingham was used for the purpose, and it is assumed that gas-water obtained from a coke-making plant would not differ much from it in composition. The above gas-water was found to contain in 100 c.c. :—

	Grms.
Ammonium chloride	0.7
Ammonium carbonate	5.2
Ammonium sulphide, Am_2S	0.4
Other compounds containing NH_3	0.3

The total of ammonia in 100 c.c. = 2.6 grms. NH_3 , equivalent to 10.0 grms. ammonium sulphate, $(\text{NH}_4)_2\text{SO}_4$.

To begin with, a sample of the gas-water was evaporated to dryness, with the loss of most of the ammonia. The residue contained only enough ammonia to make it equivalent to 1.1 grms. ammonium sulphate per 100 c.c. of gas-water.

A sample of the same original gas-water was then evaporated to dryness after having been mixed with an excess of hydrochloric acid. By this all the ammonia was

recovered. The residue was dissolved in water and boiled with sodium hydrate. The ammonia which was evolved was caused to be absorbed by a measured volume of standard hydrochloric acid. The amount of ammonia was then ascertained by titration with standard sodium hydrate solution and methyl-orange. 100 c.c. gas-water yielded 2.6 grms. NH_3 , equivalent to 10.0 grms. ammonium sulphate.

A quantity of the original gas-water was now well shaken with plaster-of-Paris; this changed all the ammonium carbonate present into sulphate, whilst a corresponding amount of the calcium sulphate was changed into carbonate. Of the clear fluid (Solution 2, sp. gr. 1.04) thus obtained, 100 c.c. were now evaporated to dryness; this operation liberated and removed a portion of the ammonia, leaving only the newly-formed ammonium sulphate and the small quantity of original ammonium chloride. By titration the quantity of ammonia present in the residue was found equivalent to 8.0 grms. of ammonium sulphate. We had thus recovered 80 per cent of the ammonia, chiefly as ammonium sulphate, without the use of sulphuric acid.

Another sample of the above-mentioned solution No. 2 was now boiled for sufficient time to expel all free ammonia, and it was arranged that this free ammonia was absorbed by standard hydrochloric acid. The ammonia absorbed per 100 c.c. solution No. 2 amounted to an equivalent of 1.7 grms. ammonium sulphate, or 17 per cent of the entire ammonia in the original gas-water. As the previous solution yielded 80 per cent, the two operations combined would yield 97 per cent of the entire quantity of ammonia. It would be possible to carry this out on a large scale. We could recover 97 per cent of the ammonia by means of gypsum, and by means of only about one-sixth of the sulphuric acid which would have been required without the use of gypsum. Solution No. 2 would first be boiled and made to yield its liberated ammonia to sulphuric acid, and afterwards it would be concentrated so as to produce crystallised ammonium sulphate.

By the use of ferrous sulphate it would also be possible, if desired, to do without sulphuric acid altogether. The gas-water would again be first treated with gypsum (or plaster-of-Paris) and the sediment of calcium carbonate carefully removed, so that the clear solution could now be treated with ferrous sulphate. The bulky precipitate of ferrous sulphide was, in our experiment, filtered off, and, after evaporating the solution, the residue yielded 95.4 per cent of the entire ammonia.

Treatment of the gas-water with gypsum, ochre, and carbon dioxide only yielded 85.7 per cent of the entire ammonia. The above-mentioned methods are thus much superior, and especially the first one with a limited use of sulphuric acid, appears most advantageous.

Birmingham, May 24, 1906.

A REVISION OF THE ATOMIC WEIGHTS OF SODIUM AND CHLORINE.*

By THEODORE WILLIAM RICHARDS
and
ROGER CLARK WELLS.

(Continued from p. 239).

THE RATIO OF SODIC CHLORIDE TO SILVER (*continued*).

THE final series of experiments, given below, includes only those experiments in which every known source of error was avoided. In order to illustrate the details of the method, the full record of a single experiment, in so far as it does not repeat what has already been described, is here given.

Experiment 53.

	Grms.
Weight of sodic chloride, in vacuum (Sample I) . . .	5.08685
Weight of silver, in vacuum (Sample O) . . .	9.38819

* Published by the Carnegie Institution of Washington, April, 1905.

These were dissolved and mixed on February 2, 1904. After two hours' agitation the mixture stood until next day, when an examination was made with the nephelometer. Two test-tubes were filled with the mother-liquor by means of a clean pipette, withdrawing 0.07 litre for this purpose. The two solutions were very nearly equal in opalescence. With occasional shaking the mixture was therefore allowed to stand yet another day, in order to ensure the thorough solution of possibly adsorbed material. The subsequent proceedings are summarised below:—

February 4 (0.07 litre withdrawn).

Time of standing in nephelometer.	Nephelometer readings.		
	(a) Reading of tube with excess AgNO_3 .	(b) Reading of tube with excess NaCl .	a + b.
Thirty minutes	45	54	0.83
Two hours	43	54	0.80
Eighteen hours	46	55	0.83

This reading indicates a slight deficiency of silver. Hence 0.15 m.grm. of silver in solution was added to the residual mother-liquor, and the mixture was occasionally shaken.

February 8 (0.070 litre withdrawn).

Time.	Nephelometer readings.		
	a.	b.	a + b.
Five minutes	48	30	1.60
Sixteen hours	60	57	1.05

There was now a slight excess of silver. Hence there was added 0.03 m.grm. of sodic chloride, corresponding to 0.06 m.grm. of silver.

February 16 (0.07 litre withdrawn).

Time.	Nephelometer readings.		
	a.	b.	a + b.
Fifteen minutes	50	39	1.28
One hour	50	44	1.14
Two hours	50	46	1.09
	53	54	0.98
	36	35	1.03
	55	59	0.93
Twelve hours	33	33	1.00
	70	67	1.04
	52	53	0.98
	37	38	0.97
	24	23	1.04
	360	362	1.00

Thus precise equality had been attained.

Volume liquor now remaining 435 c.c.
Withdrawn during trials 280 "

Original volume 715

If the first addition of Ag had been made to the original total mother-liquor it would have been $0.15 \times \frac{715}{575} = 0.19$ m.grm.

Similarly, $0.06 \times \frac{715}{505} = 0.08$ m.grm. Hence extra silver required to produce equal opalescence in original mother-liquor = $0.19 - 0.08$ m.grm. = 0.00011 gm.

Total silver = $9.38819 + 0.00011 = 9.38830$ gm. This silver contained 0.0037 per cent of impurity. Weight of pure silver present 9.38795. Then $\text{Ag} : \text{NaCl} = 9.38795 : 5.08685 = 100.000 : 54.185$.

Molecular weight $\text{NaCl} = 107.930 \times 54.185$, if

$\text{Ag} = 107.930$ 58.482
If atomic weight Cl 35.455

Atomic weight of sodium 23.027

Ten such complete analyses are recorded in the table below.

The Ratio of Sodid Chloride to Silver.

Final Series.

Exp.	Preparations. NaCl. Silver.	Weight salt (in vac.).	Observed weight silver (in vac.).	Correct weight of purest silver (vac.).	Parts NaCl equivalent to 100'000 parts silver.
		Grms.	Grms.	Grms.	
41.	F N	3'96051	7'30923	7'30896	54'187
44.	K N	2'32651	4'29371	4'29355	54'186
46.	K N	5'36802	9'90736	9'90699	54'184
45.	G M	4'00548	7'39237	7'39210	54'186
47.	G N	4'69304	8'66133	8'66101	54'186
52.	H P	3'27189	6'03864	6'03842	54'185
53.	I P	5'08685	9'38830	9'38795	54'185
55.	J P	3'66793	6'76977	6'76952	54'183
56.	L P	5'48890	10'13030	10'12993	54'185
57.	L P	3'55943	6'56933	6'56909	54'185
		41'42856	—	76'45752	54'185

The agreement of these results is now as good as could reasonably be expected. Whereas the earlier similar experiments, made before time enough had been allowed for the full development of the opalescence, showed an extreme variation in the results of 0'017, this extreme variation is now only 0'004. Since in these last experiments seven different specimens of salt were used, and three different specimens of silver, it seemed hardly probable that further work in the same direction would add certainty to the average. The chemical identity of the various preparations seems to have been proved.

The atomic weight of sodium, calculated from the ratio 100'000 : 54'185, the average of these ten determinations, is 23'027, if silver and chlorine are respectively assumed to be 107'930 and 35'455. This is 0'010 higher than the value obtained by reference to argentic chloride—a difference which seems small in comparison with much quantitative chemical work, but which, nevertheless, is far greater than the sum of the probable experimental errors of the two series of results.

The conflicting outcome of all these carefully conducted experiments thus still remained not easily explicable. Their value for the atomic weight of sodium differed by over 0'1 per cent from Stas's result, and furthermore, as has just been said, the comparison of sodid chloride with silver yielded a higher result, 23'027, than that found by comparison with argentic chloride, 23'017. Both these discrepancies required explanation before the results could be accepted without reserve.

In the first place, much time was spent in the search for some possible error in our results. For example, several of our samples of salt, especially the preparation numbered K, were tested for lithium with the greatest care by fractional treatment with alcohol. The extreme mother-liquors showed no trace of the lighter metal, although in parallel experiments it was found that less than 0'01 per cent of lithium chloride could be detected in this way, even by the simple flame test. This proportion could not affect the atomic weight of sodium by more than 0'002; but since no trace of lithium was found in any of the specimens of our salt tested, it is clear that our low value of the atomic weight could not be due to lithium. The constant results found with differently prepared samples from different sources seemed to exclude the possibility of the presence of the very few other metals which would yield too low a value for the atomic weight, namely, beryllium, magnesium, aluminium, and calcium. Acid could not have been present, since the material was usually prepared from salt finally crystallised from pure neutral solution, and was always fused in air. It gave, moreover, a wholly neutral reaction to methyl orange.

Because no flaw could be found in the operations leading to the new results, the conclusion was inevitable that the old ones were in error. In order to prove this

without question, experiments were instituted in which each one in succession of the gravest faults already pointed out in Stas's work were imitated, in order to detect the individual errors introduced by each.

In the first place, two experiments were made in which the salt was prepared approximately according to Stas's method, the substance being fused with ammoniac chlor-platinate. The salt thus prepared was compared with pure silver by means of the nephelometer, using, as Stas did, the opalescence which had been given only fifteen minutes to form. In this way 2'67137 and 5'51270 grms. of salt needed, respectively, 4'92908 and 10'1717 grms. of silver, corresponding to an atomic weight of sodium of 23'039 in each case. Since the value obtained from salt certainly free from platinum in the same way (p. 253) was 23'032, the difference of 0'007 may probably be ascribed to platinum, which had not been precipitated by thermal decomposition. Presumably, therefore, traces of platinum were present also in Stas's preparations.

In the next place, the effect of the occlusion of sodid chloride by argentic chloride was quantitatively studied. It will be remembered that Stas always dropped dry sodid chloride crystals into his silver nitrate solution instead of adding the salt in the dissolved state. Such a procedure offered the maximum opportunity for the occlusion of sodid chloride. In the following experiments, made with the purest material (salt F and silver P) and executed with all the precautions outlined above, this error alone was purposely committed. After dropping in the solid salt, the mixture, having been shaken two hours, was allowed to stand for a day, all according to the manner of Stas. In three experiments (Nos. 42, 51, 48,) 5'37853, 3'34018, and 5'29539 grms. of salt required, respectively, 9'92510, 6'16392, and 9'77122 grms. of silver, thus giving three determinations of the atomic weight of sodium, as follows : 23'033, 23'033, and 23'036, or on the average, 23'034.

In the last experiment the gradual dissolving of this occluded salt was shown in an indubitable manner. On continued shaking throughout a week, more and more silver had to be added, until finally the mixture came to complete constancy at a point corresponding to 9'77264 grms. of total silver. This quantity gives 23'028 as the atomic weight of the sodium, showing that within a week nearly if not quite all the occluded sodid chloride had been disengaged.

If, then, we sum up the effects which we have found for the three chief errors probably made by Stas in his later work, the following result is obtained :—

Our value of the atomic weight of sodium from the ratio of sodid chloride to silver	23'027
Effect of too prompt reading of opalescence	+23'005
Presence of platinum in salt	+23'007
Occlusion of salt in silver chloride	+23'007

Result which we should have obtained by Stas's complete method 23'046

This result is so near that found by Stas's assistants in 1881 (23'050) as to make it seem probable that these errors really existed in Stas's work, and that the present investigation avoided them. The acknowledged presence of traces of gas in Stas's silver and silica in his salt may be neglected in this comparison as being errors of mutually opposing tendency and almost equal effect. The rectifying of previous work by means of a later correction is always a doubtful process, and the above table is given only to show that Stas's small mistakes have all been traced.

It is not so easy to give the numerical magnitude of the correction to be applied to Stas's earlier work, which, as has been said, is inconsistent with the later work, because it gives the same result, although a wholly false end-point was used. It is probable, however, that in the early work, carried out by Stas himself, the precipitated argentic chloride was much more thoroughly shaken with its mother-liquor than the later work executed by MM. Nyst and

Cabilliau under Stas's direction. Continued shaking would diminish the solubility of the argentic chloride, as well as disengage the occluded salt. Thus the decrease of the latter error might have balanced the effect of a greater error in the end-point, although this in its turn would be less marked than it would have been had the mixture been less thoroughly shaken.

All these considerations tend to support the present determination as against the verdict of the older ones, but no explanation is yet afforded as to the reason for the difference between the values 23.017 and 23.027 found by different processes in the present work. Long consideration of the matter led finally to the conclusion that the accepted value of the atomic weight of chlorine, 35.455, upon which the calculation depends, must be in error. Accordingly this problem also was attacked experimentally. But before these lengthy experiments are recounted, brief mention must be made of the successful attack upon another doubtful point, which concerns the sodic chloride.

(To be continued).

A SYSTEM OF QUALITATIVE ANALYSIS INCLUDING NEARLY ALL THE METALLIC ELEMENTS.*

PART II.—ANALYSIS OF THE TUNGSTEN GROUP.

By ARTHUR A. NOYES.

(Concluded from p. 252).

Confirmatory Experiments and References (continued).

P. 39, N. 1.—To two portions of 7 c.c. H_2SO_4 (1.20), NH_4OH was added till alkaline, and then 7 c.c. $(\text{NH}_4)_2\text{S}$, and the mixtures were heated at $50-60^\circ$ for thirty minutes. To one of the mixtures 2 m.grms. W as Na_2WO_4 were added, and both were acidified with H_2SO_4 (1.20), and filtered; a small pink precipitate was obtained from both solutions; but the filtrate from the blank was colourless, while that from the solution containing tungsten was of a pronounced yellow colour. The experiment was twice repeated, using 1 m.grm. in place of 2 m.grms. W; a bright yellow precipitate formed on adding the H_2SO_4 , and the filtrate was distinctly yellow.

A mixture of 6 grms. of Na_2CO_3 and S with 1 m.grm. W in one case, and 6 grms. of Na_2CO_3 and S alone in another, were fused for fifteen minutes, and the fused masses dissolved in water, and the solutions filtered, acidified with H_2SO_4 (1.20), shaken, and again filtered; the mixture containing tungsten gave a yellowish pink precipitate and a slightly yellow filtrate; the blank gave a pale yellow precipitate and a colourless filtrate.

Five m.grms. W as Na_2WO_4 were treated by P. 33, 35, and 39; the precipitate was of a bright yellow colour, much deeper than that of precipitated S; the filtrate was deep yellow. The precipitate was ignited, the residue fused with Na_2CO_3 , the mass dissolved in water, and the solution boiled with HNO_3 ; a small but unmistakable, yellow precipitate of H_2WO_4 was obtained. The filtrate was treated by P. 40; a large yellow precipitate, about ten times as great as that from the H_2SO_4 precipitate, remained.

In regard to the incomplete precipitation of WS_3 by acids from solutions in alkaline sulphides, see also Jannasch and Bettges, *Ber. Chem. Ges.*, 1904, xxxvii., p. 2223.

A mixture of 300 m.grms. Sn and 2 m.grms. W was treated by P. 33 and 35, and the $(\text{NH}_4)_2\text{S}$ filtrate by P. 39; the filtrate was colourless. This filtrate was treated by P. 40; a small but unmistakable yellow precipitate of H_2WO_4 resulted.

The experiment was repeated with a mixture of 300 m.grms. Sn and 10 m.grms. W; a yellow precipitate estimated at 1 m.grm. W was obtained from the filtrate.

* From the *Technology Quarterly*, xvii., No. 3.

A mixture of 30 m.grms. Sn as SnCl_4 , 8 m.grms. W as Na_2WO_4 , and 20 m.grms. PO_4 as Na_2HPO_4 was treated by P. 35, 39, 40, 41, 43, and 44, and the HCl solution of the ignited sulphides obtained in P. 41 was divided, part being evaporated with HNO_3 , and tested with $(\text{NH}_4)_2\text{MoO}_4$, and the remainder evaporated to a small volume, diluted, treated hot with H_2S , filtered, and the filtrate concentrated and tested for tungsten with Zn; no indication of tungsten was obtained either at this last point or in P. 43 or 44; but a strong blue colour resulted at the end of P. 40. Phosphate was present in considerable quantity in the HCl solution, though the sulphide precipitate had been thoroughly washed; also, of course, in large quantity in the H_2SO_4 filtrate.

To a mixture of 200 m.grms. Mo as $(\text{NH}_4)_2\text{MoO}_4$ and 2 m.grms. W as Na_2WO_4 , and to one of 400 m.grms. Mo and 4 m.grms. W in H_2SO_4 solution, were added NH_4OH and 10 c.c. $(\text{NH}_4)_2\text{S}$, and the deep orange-red solution digested at 50° for thirty minutes, and then treated by P. 39; there resulted a black precipitate and a filtrate which was colourless with the first mixture, pale blue from the second. The filtrate was treated by P. 40; no precipitate of H_2WO_4 remained in either case. That from the second mixture was also tested with Zn and KSCN ; a pronounced but not very strong red colour resulted.

In certain test analyses, however, where molybdenum and tungsten were both present, the latter was found only in the filtrate, and there in large quantity (see T. A., 34, 36).

One m.grm. Mo and 1 m.grm. Sn in H_2SO_4 solution were separately digested with 10 c.c. $(\text{NH}_4)_2\text{S}$, and the solution acidified with H_2SO_4 ; a dark brown or yellowish brown precipitate separated, and the filtrate was colourless. The filtrate from the MoS_3 was evaporated to a volume of 3 c.c., and Zn and KSCN added; no coloration was produced.

100 m.grms. V as Na_3VO_4 were digested with $(\text{NH}_4)_2\text{S}$ as in P. 33 and 35, the small bluish grey precipitate remaining was filtered out, and the filtrate treated with H_2SO_4 by P. 39; a large brown precipitate and pale blue filtrate resulted, showing that the vanadium was divided.

P. 40, N. 3.—One m.grm. W and 50 m.grms. PO_4 as Na_2HPO_4 were added to 5 c.c. HCl (1.06) and Zn added; a pronounced blue colour resulted.

P. 41, N. 1.—The experiment first described under C. E., G. D., § 10, was repeated with 300 m.grms. W as WS_3 , with 100 m.grms. V as V_2S_5 , and with 100 m.grms. Mo as MoS_3 in separate experiments, except that the sulphides were ignited in a H_2S atmosphere, as directed in P. 41, before heating with the HCl (1.20), the crucible being heated without a jacket over a powerful burner; there was no darkening whatever of the $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$ solution in any of the three cases.

300 m.grms. Sn obtained as SnS_2 by acidifying a $(\text{NH}_4)_2\text{S}$ solution were treated by P. 41, the crucible being heated within a burnt-clay jacket for ten minutes with the full flame of a powerful burner; almost the whole mass (which was black and glistening) dissolved in the hot HCl (1.20), there being only a very small yellow residue. This residue was washed and treated with HNO_3 and HCl , the solution evaporated twice with HNO_3 , and the residue treated with HNO_3 (1.20); only a small turbidity remained.

This experiment was repeated except that the crucible was heated without a jacket and only to a low red heat; the ignited mass was mostly yellowish, but contained some black particles, and it left a large residue of yellow scales with metallic lustre when boiled with HCl (1.20).

This experiment was repeated except that the crucible was heated without a jacket to the highest temperature obtainable with a powerful burner; it was thirty minutes before the whole mass became black; after boiling with HCl (1.20) for twenty minutes, there was still a considerable quantity of a yellow scaly residue.

A mixture of 300 m.grms. Sn and 3 m.grms. W, and one of 300 m.grms. Sn and 1 m.grm. W, obtained as sulphides by adding H_2SO_4 to a $(\text{NH}_4)_2\text{S}$ solution, were

treated by P. 41 and 43; a considerable yellow residue of H_2WO_4 was obtained in the first case, and a small but unmistakable one in the second case.

200 m.grms. Sb as precipitated Sb_2S_3 were treated by P. 41 and 42; the ignited mass consisted of a black button, and this dissolved completely in the HCl (1:20) upon boiling for five minutes.

P. 42, N. 1.—One m.grm. Sn as precipitated SnS_2 was treated by P. 41 and 42, the ignition being continued for twenty-five minutes; a considerable precipitate of HgCl_2 was produced.

Separate mixtures of 300 m.grms. W as Na_2WO_4 with 1 m.grm. Sn, 2 m.grms. Sn, 4 m.grms. Sn (in two experiments), and 10 m.grms. Sn as SnCl_4 were treated with NH_4OH and $(\text{NH}_4)_2\text{S}$, and then by P. 39 and 41, and the HCl solution was then immediately added to HgCl_2 solution; no precipitate was produced in the first three cases (with 1–4 m.grms. Sn), but a fairly large one resulted in the last case (with 10 m.grms.). The experiment was repeated with a mixture of 300 m.grms. W and 1 m.grm. Sn, but the HCl solution was treated by P. 42; a distinct residue of Sn and a decided precipitate of HgCl_2 resulted.

The HCl solutions obtained in the experiments described under P. 41, N. 1, by heating on a steam-bath 300 m.grms. W as ignited WS_2 , 100 m.grms. V as ignited VS_2 , and 100 m.grms. Mo as ignited MoS_2 , with HCl (1:20), were added to HgCl_2 solution; not even a cloudiness resulted in any of the cases. The HCl solution obtained by boiling the ignited Sb_2S_3 with HCl (1:20) was added to HgCl_2 solution; a large precipitate (of SbO_3H_3) separated, but this dissolved completely upon adding HCl .

P. 43, N. 1.—300 m.grms. Mo obtained as MoS_3 by precipitation from $(\text{NH}_4)_2\text{S}$ solution by H_2SO_4 were boiled with 10 c.c. HNO_3 (1:10); apparently all the molybdenum went into solution. The residue was washed and treated repeatedly with a portion of NH_4OH ; the solution was acidified with HCl , and treated with Zn and KSCN ; no red coloration appeared.

A mixture of 2 m.grms. W and 500 m.grms. Mo in HF solution was treated by P. 3, 5, 6, 33, 35, 39, and 43; an unmistakable precipitate of H_2WO_4 was obtained.

100 m.grms. Mo and 100 m.grms. V as ignited sulphides were separately warmed on a steam-bath with 8 c.c. HNO_3 (1:42) and 2 c.c. HCl (1:20); in two minutes the residue dissolved completely. The molybdenum solution was evaporated to 3 c.c.; a large precipitate separated.

The experiment was repeated with 300 m.grms. W as ignited WS_2 ; the black residue was completely converted into a yellow one within five minutes.

In regard to the difficulty of separating H_2WO_4 completely by the addition of strong acid to a tungstate solution, see Jannasch and Bettges, *Ber. Chem. Ges.*, 1904, xxxvii., p. 2225.

P. 43, N. 2.—Mixtures of 2 m.grms. W as Na_2WO_4 and 100 m.grms. Mo as $(\text{NH}_4)_2\text{MoO}_4$ (in two experiments), and of 4 m.grms. W and 100 m.grms. Mo were treated by P. 35, 39, 40, 41, and 43; no precipitate of H_2WO_4 was obtained in any case from the H_2SO_4 filtrate tested by P. 40; and no precipitate separated from the HNO_3 and HCl solution of the ignited sulphides even on standing over-night; but upon evaporating this solution with H_2SO_4 , a large yellow residue separated from the mixture containing 4 m.grms. W, an extremely small but distinct one from one of those containing 2 m.grms. W, and nothing whatever from the other containing 2 m.grms. even on standing over-night.

P. 44, N. 1.—0.5 m.grm. W as Na_2WO_4 was added to 2 c.c. HCl (1:12) and a few granules of zinc introduced; a strong blue coloration appeared almost immediately, and in a few minutes blue flocks separated from the liquid. The colour persisted for at least fifteen minutes. The experiment was repeated with 2 m.grms. W; an intensely blue flocculent precipitate separated, and on filtration the mixture yielded a perfectly colourless filtrate.

To 10 m.grms. Mo and 100 m.grms. Mo as $(\text{NH}_4)_2\text{MoO}_4$,

dissolved in a few drops of water, 2 c.c. HCl (1:12) and a few granules of zinc were added; in each case the solution became yellow at once, then dark orange-yellow, next dark green, and finally dark olive-brown, but no precipitate formed except after long standing; at no time was a blue coloration noticeable.

100 m.grms. Mo as $(\text{NH}_4)_2\text{MoO}_4$ were dissolved in 10 c.c. water and 0.5 c.c. HCl (1:12) added; the solution soon assumed a clear blue colour, which became deep blue after some minutes.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, May 17th, 1906.

Prof. R. MELDOLA, F.R.S., President, in the Chair.

MESSRS. E. Barrett, E. R. Chrystall, J. L. Foucar, P. H. Marsden, H. Martin, J. T. Nance, W. D. Seaton, W. H. Simmons, D. Sommerville, P. F. Spencer, F. W. Storey, R. W. Tonkin, and J. A. Watson were formally admitted Fellows of the Society.

Certificates were read for the first time in favour of Messrs. Shelton Gottlieb Agar, Panorama House, Guernsey; Thomas William D. Gregory, 82, Moorland Road, Burslem; John Gerard Hughes, 2, Canute Road, Southampton; Ernest Arthur Jenkinson, Dauntsey Agricultural School, West Lavington; William Rest Mummery, The Firs, Shenfield, Essex; Arthur Charles Palmer, B.Sc., 17, Wansbeck Gardens, West Hartlepool; Samuel Shrowder Pickles, B.Sc., The Imperial Institute, S.W.; Willie Macro Seaber, B.Sc., Firdale, Sheen Lane, East Sheen; James Neil Watts, P.O. Eikenhof, Johannesburg, S. Africa.

Certificates have been authorised by the Council for presentation for ballot under By-law I. (3) in favour of Surendra Prasad Sanyal, M.A., Majhowli Raj, Gorakhpur, U.P., India; Allan Sime, Kingston, Jamaica; Edward Jocelyn Wortley, Rectory, Half-way Tree, Jamaica.

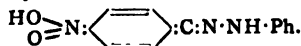
Of the following papers, those marked * were read.

*98. "The Relation between Absorption Spectra and Chemical Constitution. Part VI. The Phenylhydrazones of Simple Aldehydes and Ketones." By EDWARD CHARLES CYRIL BALY and WILLIAM BRADSHAW TUCK.

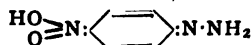
A spectroscopic investigation of the phenylhydrazones of formaldehyde, acetaldehyde, propylaldehyde, acetone, and diethylketone shows that all these compounds exist in two forms, the true hydrazone and the azo-form, in which the hydrogen atom has wandered from the nitrogen atom to the carbon atom of the aldehyde or ketone residue, thus: $\text{Ph}\cdot\text{NH}\cdot\text{N}\cdot\text{CH}\cdot\text{CH}_3$ and $\text{Ph}\cdot\text{N}\cdot\text{N}\cdot\text{CH}_2\cdot\text{CH}_3$. The azo-form is the more stable, and all the above compounds readily pass over into this form under the influence of light, except formaldehyde phenylhydrazone, which polymerises. All the azo-compounds are strongly coloured, owing to the isorropesis between the unsaturated nitrogen atoms and the benzene nucleus. If the compounds are prepared from *p*-bromophenylhydrazine, the change to the azo-form takes place quite slowly; when aldehydes and ketones are allowed to react with *p*-bromophenylhydrazine, the true hydrazone is always produced first, which is not always the case when phenylhydrazine is used, for in certain instances the azo-compound is obtained at once. As is to be expected, true hydrazones which are perfectly stable are produced by the interaction of aldehydes and ketones with phenylmethylhydrazine.

An investigation into the absorption spectra of the hydrazones of the three isomeric nitrobenzaldehydes shows that the colour of these substances is not due to their existence in the azo-form. It is found, however, that they exist in the quinonoid form and are somewhat similar to

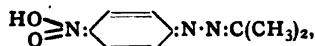
the nitrophenols. The formula of the *para*-compound is, in all probability,—



Similarly, in the case of *p*-nitrophenylhydrazine and its acetone derivative, these compounds are quinonoid, thus:—



and—



and are comparable with *p*-nitroaniline.

DISCUSSION.

Mr. W. ROBERTSON asked Mr. Baly if he had examined the spectra of the two pure acetaldehydephenylhydrazones individually, inasmuch as the product of melting-point 80° , found by Mr. Baly to give rise to the absorption band in the ultra-violet, is far from being a pure substance. It would be of considerable interest to ascertain if this band is also shown by either isomeride in the pure state in neutral solvents.

Dr. LANDER asked the authors whether the change of hydrazone to azo-compound was complete, or whether the two substances formed a mixture in equilibrium in solution.

Dr. HEWITT asked Mr. Baly if he had examined any hydrazones obtained from quinolyhydrazines. *Ortho*- and *ana*-quinolyhydrazines were examined some years back by Dufton (*Trans.*, 1891, lix., 756; 1892, lxi., 782), when it was found that certain of the derived hydrazones, more especially those prepared with *ana*-quinolyhydrazine, exhibited brilliant colours. The author did not, however, suggest for them any other than a hydrazone structure (compare Armstrong, *Trans.*, 1892, lxi., 789).

Mr. BALY said that the two isomerides mentioned by Mr. Robertson would, if present, give identical absorption bands. He hoped to extend the work in very many directions, but had not yet examined the substances mentioned by Dr. Hewitt.

*99. "The Rusting of Iron." By JOHN TRENGROVE NANCE.

It is known that ammonium chloride solution accelerates the rusting of iron. The author finds that interaction takes place with evolution of hydrogen and liberation of ammonia; iron passes into solution in the ferrous state, and is not precipitated in the absence of air, owing to the excess of ammonium chloride present.

The solubility of iron in solutions of different concentration does not diminish proportionally with the amount of ammonium chloride present; hence it is probably due to the action of hydrogen ions formed by hydrolysis of the salt, as similar proportional numbers are found for the solubility of iron and for the catalytic effect of the solutions on the hydrolysis of methyl acetate. The rate of rusting of iron in these solutions varies with the concentration in a similar manner, leading to the conclusion that rusting is due to the (mainly catalytic) action of hydrogen ions. In support of this, it is found that the chlorides of weak bases accelerate rusting far more than do those of the strong bases, and that the influence of acids is roughly proportional to their avidities.

DISCUSSION.

Dr. MOODY thought it unfortunate that chemists were not definitely agreed as to the exact meaning to be attached to the term rusting. It appeared desirable that the term should be limited to those changes which metals undergo on exposure to air under normal atmospheric conditions. With regard to the results obtained by the author of the paper under discussion, it must be borne in mind that on heating a solution of ammonium chloride at 80° ammonia would be evolved, and free hydrochloric acid would be formed in solution. In fact, the experiments resolved

themselves into a determination of the attack on iron by solutions of hydrochloric acid of different strengths, and the theoretical conclusions drawn by the author appeared unwarranted.

Dr. F. M. PERKIN agreed with Dr. Moody that the term rusting should be used to denote the atmospheric oxidation of iron, and should not be employed in reference to oxidation by other agencies.

He pointed out that many other metals were dissolved by ammonium salts, and asked whether Mr. Nance had tried the action of ammonium persulphate, which dissolved iron much more rapidly than other ammonium salts. The solution of the metals was evidently due to hydrolysis, and, as ferric salts are much more readily hydrolysed than ferrous salts, the explanation of the rapid solution in ammonium persulphate was to be traced to the conversion of the ferrous salt into the ferric condition. Oxidation of iron might be brought about by any feebly ionised acid. With highly ionised acids, as long as excess of acid is present, no precipitation of hydroxide will take place, but with excess of iron, hydrolysis will ultimately ensue.

In reply, Mr. NANCE said that he considered his results sufficient to prove that atmospheric corrosion and rusting of iron takes place through the catalytic intervention of hydrogen ions.

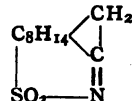
*100. "Aromatic Compounds obtained from the Hydro-aromatic Series. Part II. The Action of Phosphorus Pentachloride on Trimethylidihydroresorcin." By ARTHUR WILLIAM CROSSLEY and JAMES STUART HILLS.

When phosphorus pentachloride acts on trimethylidihydroresorcin, two substances are produced, a liquid boiling at $118-119^\circ$ at 33 m.m., which is 3:5-dichloro-1:1:2-trimethyl- Δ^2 :4-dihydrobenzene, and a solid melting at 76.5° , namely, 3:5-dichloro-1:2:6-trimethylbenzene. This aromatic dichloride is converted on oxidation into 3:5-dichlorohemimellitic acid, crystallising from water in transparent, hexagonal plates, containing $2\text{H}_2\text{O}$, and melting at $226-227^\circ$, at which temperature it is rapidly converted into its anhydride. The trimethyl ester crystallises in rhombohedra melting at $62-63^\circ$, and the monomethyl ester melts at $141-142^\circ$.

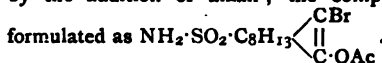
*101. "Studies of Dynamic Isomerism. Part V. Isomeric Sulphonic Derivatives of Camphor." By THOMAS MARTIN LOWRY and EGBERT H. MAGSON.

Measurements were given of the solubility of a series of twenty sulphonic derivatives of camphor, both alone and in presence of a trace of alkali. The majority of the compounds derived from α -bromocamphor and α -chlorocamphor showed an increase of solubility similar to that observed in these compounds themselves, and in their β - and π -halogen derivatives (Lowry, *Proc.*, 1906, xxii., 70).

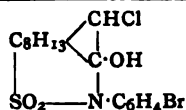
In the case of camphor sulphonamide, a decrease of solubility resulted from the addition of alkali, and it was found that the amide, which crystallises unchanged from acetic anhydride, had been converted quantitatively by N/500 sodium ethoxide in alcoholic solution at 20° into the anhydramide,—



The π -amide derived from α -bromocamphor could not be converted into an anhydramide, but was found to yield an acetyl derivative, the solubility of which was unchanged by the addition of alkali; the compound is therefore

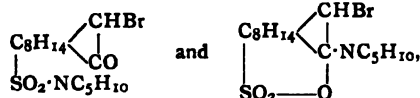


The β -sulphonanilides derived from α -bromocamphor and α -chlorocamphor dissolve readily in caustic soda and are re-precipitated by acids. Possibly on account of their acid properties, the anilides, which may be formulated as,



do not increase in solubility when a trace of alkali is added.

The isomerism previously observed in the camphor sulphopiperidides (Armstrong and Lowry, *Trans.*, 1902, lxxxii., 1449) has now been detected in the α -bromopiperidides; both compounds, formulated as—



increase in solubility when alkali is added, and probably exist in stereoisomeric forms.

*102. "The Densities of Liquid Nitrogen and Liquid Oxygen and of their Mixtures." By JOHN KENNETH HAROLD INGLIS and JOSEPH EDWARD COATES.

The authors have determined the densities of liquid nitrogen and of liquid oxygen, and of a number of mixtures of the two liquids at the temperatures 74.70° and 79.07° Abs. on the hydrogen scale. These are the temperatures at which the vapour pressure of pure oxygen is 100 m.m. and 200 m.m. respectively, and they are therefore easily reproduced. The results showed that a slight contraction took place on mixing the two liquids, this contraction being greater at the higher temperature. The densities of the pure liquids differed by about 1 per cent from the values obtained by Baly and Donnan (*Trans.*, 1902, lxxxi., 907). These values of the densities of the mixtures may be used to determine the relation between the partial pressures of nitrogen and oxygen above a mixture of the two liquids and their concentrations in that mixture. It is found that the solubility of nitrogen in oxygen obeys Henry's law; but that the solubility of oxygen in nitrogen does not obey the simple form of that law, for oxygen dissolved in nitrogen is associated to the extent of about 9 per cent.

103. "Glutaconic and Aconitic Acids." By HAROLD ROGERSON and JOCELYN FIELD THORPE.

Ruhemann is mistaken when he states (*Proc.*, 1906, xxii., 137) that we were not fully acquainted with his several publications dealing with these acids.

When we recorded the reactions of γ - and $\beta\gamma$ -aliphyl substituted derivatives of 2:6-dioxypyridine (*Trans.*, 1905, lxxvii., 1682), we compared them with the corresponding reactions of the aliphyl β -substituted derivatives prepared by Ruhemann (*Trans.*, 1893, lxiii., 876). The γ - and $\beta\gamma$ -derivatives prepared by him (*Trans.*, 1899, lxxv., 245), and which he complains have not been mentioned by us, are aryl derivatives containing phenyl and benzyl groups in these positions, and therefore were not, in our opinion, suitable for the purpose of comparison. As a matter of fact, the behaviour of our aliphyl compounds differs in many important respects from that of the aryl compounds prepared by him.

Ruhemann has also evidently misunderstood our meaning, since he writes (*loc. cit.*):—"They state that ethyl dihydroxycinchomeronate had first been prepared by Errera and Perciabosco, but they overlook the fact that Ruhemann and Stapleton had obtained it the year before." The passage referred to (*Trans.*, 1906, lxxxix., 640) is:—"It is evidently identical with the compound prepared by Errera and Perciabosco by the action of hydrochloric acid on ethyl 2:6-dihydroxypyridine-3:4:5-tricarboxylate." We did not desire to assign priority to Errera and Perciabosco, but merely wished to establish the identity of our compound, an object which seemed to us better attained by referring to the method of preparation adopted by these chemists, rather than to the more indirect process used by Ruhemann and Stapleton.

We have carefully re-read the paper by Ruhemann and Orton (*Ber.*, 1894, xxvii., 3449), and are still of the opinion that the remark (*Trans.*, 1906, lxxxix., 634), "Ruhemann and Orton suggest that aconitic acid corresponds to fumaric acid in constitution, whereas the malenoid form is represented by aceconitic acid," is not an overstatement of the case, since probability surely implies suggestion.

Regarding the criticism of our formulae for these acids and the remark, that if they were correct, then the action of ammonia on ethyl aconitate might be expected to yield a five-membered ring compound besides citrazinamide, we cannot too strongly emphasise the remarks we made in our reply to Feist and Beyer (*Trans.*, 1906, lxxxix., 650), which were to the effect that our formulae for these compounds are merely an effort to represent diagrammatically a state of equilibrium between two tautomeric forms.

104. "The Chemistry of Organic Acid 'Thiocyanates' and their Derivatives." By AUGUSTUS EDWARD DIXON.

Compounds of the class R·CO(CNS), when reacting with primary nitrogenous bases, present marked differences of behaviour, according to the nature of the hydrocarbon radicle R. If R be aromatic, these compounds behave in ordinary circumstances principally as thiocarbimides, uniting additively with the base employed to form in each case a symmetrically disubstituted thiocarbamide, R·CO·NCS + R'·NH₂ = RCO·NH·CS·NHR'. On the other hand, if R be a fatty radicle, the function of the associated group, —CO(CNS), varies greatly with the conditions—notably temperature and the nature of the base presented. In such cases, the end-products contain both disubstituted thiocarbamide and a mixture of substituted amide with thiocyanic acid, the latter substances originating through double decomposition: R·CO·SCN + R'·NH₂ = R·CO·NHR' + H·SCN.

The interposition of an oxygen atom between the radicle R and the group CO(CNS), where the former belongs to the aliphatic series, increases the thiocarbimide power of the resultant molecule; thus, phenacetyl "thiocyanate," Ph·CH₂·CO(CNS), and phenoxyacetyl "thiocyanate," Ph·O·CH₂·CO(CNS), readily yield the reaction for thiocyanic acid, whilst carbo-benzyloxy "thiocyanate," Ph·CH₂·O·CO(CNS), does so to a very trifling extent.

A number of substituted thioureas and thiocarbamides are described, resulting from the action of ammonia or other bases on various thiocarbimides, RO·CO·NCS, where R = phenyl, o-tolyl, p-tolyl, and benzyl respectively.

When acetylphenylbenzylthiourea was heated under diminished pressure, a liquid distilled over having the general properties of acetyl "thiocyanate"; this fact is regarded as confirmatory of Hawthorne's view (*Trans.*, 1906, lxxxix., 566) that acetylthiocyanate is identical with acetylthiocarbimide.

Phenylchlorocarbonate united directly with thiourea to form a compound of formula NH:C(NH₂):S·CO·OPh, HCl; on treating with dilute alkali, diphenyl carbonate and thiourea were obtained, and not the corresponding base. Unlike the fatty derivatives obtained from methyl or ethyl chlorocarbonate, the hydrochloride, when decomposed by heating, did not yield a pseudo-thiourea, NH:C(NH₂):S·Ph.

105. "The Molybdilactate and the Tungstilactate of Ammonium." By GEORGE GERALD HENDERSON.

The observations recorded in a previous paper (*Trans.*, 1903, lxxxiii., 259) on the influence of molybdic and tungstic anhydrides on the specific rotations of solutions of alkali l-lactates pointed to the conclusion that compounds of the type RO₂(C₃H₅O₃M)₂ are formed both when the respective anhydrides are dissolved in hot solutions of the alkali lactates and when alkali molybdates and tungstates are heated with solutions of lactic acid. Attempts have since been made to isolate the molybdi- and tungstilactates so produced, only one of which, namely, potassium molybdilactate, had already been obtained in crystalline form, and then in very small quantity and in a slightly impure state (*Trans.*, 1899, lxxv., 554). The results were disappointing so far as the metallic salts were concerned,

but, on the other hand, well-defined stable ammonium salts of molybdilactic and tungstilactic acids were easily obtained. Thus, finally, crystalline derivatives of the tartar emetic type have been prepared from all the hydroxy-acids which were selected for examination, and in the latter case, as in the others, the author's suggestions regarding the constitution of these compounds are supported by the results of experiment.

Molybdic and tungstic anhydrides are dissolved, the latter with some difficulty, when heated on the water-bath with solutions of alkali lactates. The reaction comes to an end when the substances are present in the proportion of one mol. anhydride to two mols. lactate, and no reduction of the anhydride occurs unless a trace in excess of that amount is added, provided that the solution is not heated to boiling. Addition of alcohol to the concentrated solutions causes the separation of colourless viscous liquids which do not become crystalline even on prolonged contact with the mother-liquor. When left in a vacuum desiccator over sulphuric acid, the syrupy liquids slowly dry to amorphous glassy masses, which are extremely deliquescent, very readily soluble in water, and fairly so in dilute alcohol. Owing to the failure of all attempts to induce these substances to crystallise, they could not be obtained in a satisfactorily purified state, but an analysis of the compound of molybdic anhydride and sodium lactate, partially purified by dissolving in a little water, and precipitating with alcohol, showed that its percentage of sodium was only a little higher, and of molybdenum a little lower, than that required by the formula $\text{MoO}_2(\text{C}_3\text{H}_4\text{O}_3\text{Na})_2$. Similar results were obtained by experiments with calcium and barium lactates. The anhydrides are dissolved fairly readily by hot solutions of these salts, but the compounds formed could not be isolated except as vitreous solids, which analysis proved to be somewhat impure.

The results were different when ammonium lactate was used. Molybdic anhydride was readily dissolved when heated on the water-bath with a solution of the salt, and, as in the case of the metallic lactates, no reduction took place unless a greater proportion of the anhydride than one mol. to two mols. lactate was added. After heating for some time, the solution was concentrated to a small bulk, and, after cooling, a quantity of a crystalline salt separated. The salt was purified by crystallisation from water, dried in a desiccator over sulphuric acid, and analysed:—

1.0 gave 0.097 NH_3 . $\text{NH}_3 = 9.7$.
0.5 „ 0.235 MoS_2 . $\text{Mo} = 28.2$.
 $\text{MoO}_2(\text{C}_3\text{H}_4\text{O}_3\text{NH}_4)_2$ requires $\text{NH}_3 = 10.0$; $\text{Mo} = 28.2$ per cent.

Ammonium molybdilactate, $\text{MoO}_2(\text{C}_3\text{H}_4\text{O}_3\text{NH}_4)_2$, forms small colourless crystals which are fairly readily soluble in water and insoluble in alcohol. It is stable in the dry state at the ordinary temperature, but undergoes decomposition with loss of ammonia when heated in a steam-oven. It is also stable in aqueous solution, but is hydrolysed on warming with dilute alkalis or mineral acids. When silver nitrate is added to a solution of the salt, a yellow precipitate is formed which quickly turns black if exposed to light.

Ammonium tungstilactate, $\text{WO}_2(\text{C}_3\text{H}_4\text{O}_3\text{NH}_4)_2$, was prepared and purified in a similar manner. It is a colourless, crystalline salt, closely resembling and apparently isomorphous with the corresponding molybdilactate, than which it is somewhat more stable, as it does not begin to decompose when heated until a temperature of about 120° is reached, when ammonia is evolved. It dissolves in water fairly readily and without change. Analysis of the purified salt, dried at 100° , gave the following results:—

0.5 gave 0.406 NH_3 . $\text{NH}_3 = 8.12$.
0.5 „ 0.268 WO_3 . $\text{W} = 42.5$.
 $\text{WO}_2(\text{C}_3\text{H}_4\text{O}_3\text{NH}_4)_2$ requires $\text{NH}_3 = 7.94$; $\text{W} = 42.9$ per cent.

My thanks are due to Mr. Hugh B. Brown for assistance in this work.

PHYSICAL SOCIETY.

Ordinary Meeting, May 25th, 1906.

Dr. C. CHREE, F.R.S., Vice-President, in the Chair.

A PAPER ON "Colour Phenomena in Photometry" was read by Mr. J. S. Dow.

The question has often been raised whether it is physiologically possible to compare lights of different colours. The author has found that it is chiefly a matter of practice. A great difficulty arises from the fact that the central portion of the retina is more sensitive to red, and less sensitive to green, than the surrounding portion. Therefore, when we attempt to photometer lights of different colour, enormous differences are found as the distance of the eye from the photometer-screen is altered, and utterly different results are obtained with different photometers. Curves are given in the paper showing the effect of altering the distance of the eye when an incandescent mantle is compared against a gas standard with the Joly photometer. Differences of 5 per cent. can easily be obtained. An experiment was shown at the meeting to demonstrate that the Purkinje Phenomenon, generally regarded as a great cause of uncertainty in ordinary work, only becomes noticeable at very small illuminations and with comparatively large fields of view. The Purkinje effect can be physiologically explained by the action of the "rods" and "cones" on the retina. The comparative weakness of the Purkinje effect with small fields can be explained by the fact that the central portion of the eye contains practically no rods but only cones.

The paper also describes experiments to show that Flicker photometers seem to be affected by colour-phenomena, but to a smaller extent than ordinary ones.

In any case, whether Flicker or an ordinary photometer is adopted, it is necessary to specify the size of the field, the distance of the eye, and the order of illumination used, in order to get consistent results.

Mr. A. P. TROTTER expressed his interest in the paper, and said he thought the difficulty of comparing the intensities of two lights of different colours was an imaginary one. It was possible for anyone accustomed to photometric measurements to get reliable comparisons of lights of different colours. The author, for the sake of precision, had dealt with the comparison of red and green, but he pointed out that in practice the variation in colour between the lights under test was not so great as this; ranging from the light of the Hefner lamp to that of the arc-light or daylight. He agreed with the author that when taking photometrical measurements it was best to get as far away as possible from the comparison disc.

Mr. A. RUSSELL considered Mr. Dow's explanation of the phenomena of heterochromic photometry by means of the irregular distribution of rods and cones on the retina quite satisfactory. Whatever theory we adopt as to the difference between the functions of the rods and cones, it is obvious that their relative numbers on the portion of the retina covered by the image of the photometer-disc is a factor of primary importance in determining the balance. It is a physiological fact that there are no rods on the yellow spot, and this had led Professor Blondel to suggest that if we arranged so that the image always fell on the yellow spot, consistent results might be obtained. Reference was also made to M. Lauriol's paper in the *Bulletin* of the Société Internationale des Electriciens on the effect of the speed of the rotating part of the Flicker photometer on the balance obtained. The experiments proved that many of the results obtained depended merely on the speed of rotation. When comparing green with red, the numbers obtained varied between the wide limits of 1 to 5. Even when comparing a Nernst with an

ordinary glow-lamp, a variation of 20 per cent with speed was noticed. It was pointed out that it was easier to obtain a balance when comparing red with green by using a grease-spot photometer than by using a Lummer-Brodhun contrast photometer. The colours of the two sides of the grease-spot in the position of balance, owing to the transmitted light, were not pure red and green, but were mixtures which it was much easier to compare. The experience of the speaker was that the Lummer-Brodhun photometer was the one most favoured by those engaged in accurate photometric work. For comparing lights of different colours, however, a modified form of grease-spot photometer would have many advantages.

Mr. S. SKINNER described an Automatic Arc-lamp, exhibited by Mr. H. Tomlinson and Rev. G. T. Johnston.

This is a simple form of automatic arc-lamp which can be constructed by any amateur at small expense. A vertical brass tube supported by a wooden framework carries the upper carbon, which can be raised or lowered by hand and clamped in any position in the tube. The lower carbon fits into a hollow brass tube, and into the lower part of the tube is fitted an iron plunger. The plunger is surrounded by a solenoid, consisting of a single layer of No. 14 copper wire, the internal diameter of the solenoid being slightly greater than the diameter of the plunger. The plunger dips into a small box of mercury, and is made to float upright by means of a brass collar and by the rounded ends of three nails forming an equilateral triangle. The current enters the upper carbon through the brass cylinder, passes through the lower carbon into the mercury, and then through the solenoid. To "strike the arc" the lower carbon is raised by hand to touch the upper one, and the plunger is then permitted to sink into the mercury until the suction of the solenoid balances the buoyancy of the mercury. With currents from 2 to 6 amperes the light is very steady, but for currents above 4 amperes it is suggested that the solenoid should have two layers of wire arranged in parallel. For currents less than 3 amperes these layers could be arranged in series.

A paper on "*The Theory of Moving Coil and other kinds of Ballistic Galvanometers*" was read by Prof. H. A. WILSON.

In this paper the exact formulæ giving the quantity of electricity passed through various types of ballistic galvanometers in common use are obtained. It is shown that the different types require different formulæ, all of which, however, reduce to the same formula when the angle of deflection is very small. In the case of a moving-coil galvanometer having a rectangular coil, iron core, and pole-pieces arranged so as to give a radial magnetic field, the formulæ take a simple and convenient form.

Mr. A. CAMPBELL exhibited a Bifilar Galvanometer free from Zero Creep.

For measuring direct currents and voltages of ordinary range (from 0.1 ampere or volt and upwards) moving-coil galvanometers with shunts or added resistances are convenient. The usual instruments, however, are affected by gradual displacement of zero (due to elastic set) when a deflection is maintained for some time. This difficulty is got over by replacing the ordinary torsional suspension by a bifilar system in which the two wires are so far apart that the gravity control quite swamps that due to the torsion of the wires. In the instrument shown, the wires are more than 1 c.m. apart, and the sensitivity with 40 ohms resistance is 400 μ m. at 1 metre for 0.001 ampere; this is sufficient for many purposes. The full deflection may be maintained for hours without causing a zero creep of 1 part in 2000. To attain good damping a very powerful magnet is used. The arrangement of this and the general design are due to Mr. R. W. Paul, who has adapted to the purpose Mr. Evershed's system of coil and magnet.

Prof. H. A. WILSON asked if the zero creep might not be due to magnetic material in the coil. If the field was

unsymmetrical the presence of magnetic material would produce a creep.

Mr. CAMPBELL replied that in very sensitive moving-coil galvanometers the presence of magnetic dirt was a source of trouble, but in the instrument which he had exhibited the control was so strong that the creep due to this cause was practically nothing.

SOCIETY OF CHEMICAL INDUSTRY. (LONDON SECTION).

Ordinary Meeting, May 21st, 1906.

Mr. A. GORDON SALAMON in the Chair.

"The Electro-Chemical Problem of the Fixation of Nitrogen." By Prof. PHILIPPE A. GUYE.

The author demonstrates the importance of the problem of the fixation of nitrogen by recalling the principal statistics relative to the total consumption of Chilean nitrate and of ammoniacal salts, and by referring to their principal chemical and agricultural uses.

Among the many investigations undertaken to solve this problem two directions have led to industrial methods—the one, calcium cyanamide; the other, electrochemical nitric acid.

The principal technical details of the manufacture of calcium cyanamide are given, and it is pointed out that its cost price depends upon that of calcium carbide. From this it is concluded that a kilogram of nitrogen fixed as calcium cyanamide will cost a little more than ammonia salts and Chili saltpetre, if the excess of calcium carbide obtained in carbide works not available for the development of acetylene is used. This conclusion seems confirmed by some agricultural tests made with this new compound of nitrogen of which the value, relative to Chili saltpetre, is not definitely fixed.

Passing to electrochemical nitric acid the author summarises the principles of its preparation, and although these are very simple, the application has presented serious difficulties, which, however, now appear to be solved by the experiments carried out in Norway. The absorption of the nitric acid by sulphuric acid is insisted on, as this allows concentrated nitric acid to be directly obtained, which is of greater commercial value than nitrate of lime, and consequently of more interest to a new industry. Analysis of the cost of electro-chemical nitric acid leads to the conclusion that a kilogram of nitrogen is fixed slightly cheaper as nitric acid than as calcium cyanamide.

In concluding, the author discusses the exterior economic factors which may hasten the development of the nitrogen industries. Among these the direct synthesis of ammonia from nitrogen and hydrogen, and the recovery of the nitrogen of coal in the ammoniacal form by the methods of L. Mond, are mentioned. These processes, combined with the production of electro-chemical nitric acid, will in all probability solve the solution of obtaining electric energy cheaply by motors utilising the power of coal.

THE SIXTH INTERNATIONAL CONGRESS OF APPLIED CHEMISTRY.

The Committee responsible for the arrangements made for the Sixth International Congress of Applied Chemistry may congratulate themselves upon having organised a most successful meeting. The Italian Government placed at their disposal the new Palace of Justice, which building offered every advantage for the purposes of such a Congress. On the evening preceding the formal opening by the King and Queen of Italy on April 26th, a pleasant social gathering of the members of the Congress took place at the Hotel Excelsior, on the invitation of the Chemical Society of Rome. The meetings of the sixteen

sections into which the Congress was divided, occupied both the mornings and afternoons from April 26th to May 3rd, while general meetings were held on April 28th to hear Sir William Ramsay's paper dealing with sewage disposal; on the 30th, when Professor Moissan read a highly interesting paper on the distillation of the metals; on May 1st, when Dr. Adolf Frank described his process for the utilisation of the nitrogen of the atmosphere for the preparation of artificial manures; and on May 2nd, when Dr. Otto Witt explained the objects of the Congress and the true meaning of the term "Applied Chemistry." On May 5th, a State banquet was held for the delegates of foreign governments and societies, and a reception by the King at the Quirinal. The Committee had arranged many social gatherings and excursions to places of interest in the neighbourhood, and these were largely attended by the members, who were enthusiastic about the scenic beauties of such places as Tivoli, and the artistic treasures of Rome itself. Circumstances arose to prevent some of the longer excursions to Sicily and Elba. It is no doubt unnecessary to emphasise the fact that the utmost hospitality was extended to the 1800 members of all nationalities who attended the Congress, and that every possible arrangement was made for the comfort and welfare of their guests by the Committee. The seventh meeting is to take place in London in 1909, on the invitation of Professor Tilden, representing the British Government, and of a Joint Committee elected by the members of the various British societies connected with chemistry, and special interest will no doubt be evinced in it, since it will be the first meeting to be held in London.

Among the more important papers read before the Congress may be specially mentioned Sir William Ramsay's excellent account of the purification of sewage water, in which he discussed the different methods now employed for this purpose, laying stress on bacteriological processes.

Professor Moissan's description of his work on the distillation of the metals was also heard with much interest. By means of the electric furnace he has succeeded in distilling copper with great ease, gold being somewhat more refractory than copper, while the platinum metals are still more difficult to distil. The metals of the iron group are vaporised at the temperature of the furnace, and also boron, carbon, titanium, and silicon. Thus the author has now shown that all substances are volatile at about 3500°, and this is therefore the highest limit of the temperature of the sun, which must in reality lie some hundreds of degrees lower than this limit, as most of the elements of which it can be shown by spectral analysis to consist are volatile at a lower temperature.

Sir William Ramsay contributed also a paper on the Bischoff process for manufacturing white lead, which does away with the dust which constitutes a danger to the work-people. In this process litharge is reduced at about 300° to a black lead suboxide of variable composition, by means of water-gas, prepared by leading a current of water vapour over strongly heated coke. The suboxide is mixed with water till a yellow mixture of oxide and hydroxide is obtained; this is saturated with carbon dioxide and a dilute solution of lead acetate added, the white lead thus obtained being compressed and mixed with oil.

Two papers by C. Nicolardot, read before the Analytical Chemistry Section, describe methods of separating iron from metals and metalloids, which seem likely to find practical application. The process in its simplest form is carried out as follows:—The alloy or ore is first dissolved by means of hydrochloric acid or nitric acid, or a mixture of these acids, the solution is filtered and oxidised with nitric acid or chloric acid. If no volatile chlorides are present the solution is evaporated to dryness, or if aluminium is present it is merely heated to 100°. After standing for some hours it is dissolved, the liquid is neutralised with ammonia if it contained volatile compounds. It is then boiled, ammonium sulphate solution added, and the precipitate which slowly forms is filtered and well washed. If chromium or aluminium is present it

is best to treat the ore with hydrochloric or sulphuric acid, or to fuse with acid potassium sulphate, filter, evaporate to dryness, re-dissolve the residue, and add SO₂ solution; the chromium and aluminium sesquioxides are then separated off, the liquid is neutralised in the cold with ammonia, and ammonium sulphite is added, the solution boiled, and the basic sulphites ignited.

Professor Garelli and Dr. Barbieri in an interesting paper described the preparation of thorium and cerium from monazite sand. The process consists in heating the sand with sodium peroxide, or caustic soda and potassium chlorate, dissolving in sulphuric acid, and treating the solution with ammonia. The precipitate thus obtained is then treated with oxalic acid, and the cerium separated from thorium either with thiosulphate or with salicylic acid.

A paper which was of considerable practical importance was that by Dr. Adolf Frank on the utilisation of the nitrogen of the atmosphere for the production of manures and other nitrogenous substances. Air is first liquefied and fractionally distilled, and thus the nitrogen is separated. This is then heated with calcium carbide, and the calcium cyanamide thus produced may be used directly as a manure. The oxygen obtained as a by-product may be used for the preparation of nitric acid from the ammonia which is also formed from the cyanamide. By means of this process the author has prepared the following substances:—Pure compressed ammonia, ammonium salts, urea, dicyanamide and its salts, salts of dicyandiamidine and of guanidine, artificial indigo, &c., so that the great technical importance of Dr. Frank's discovery will at once be recognised.

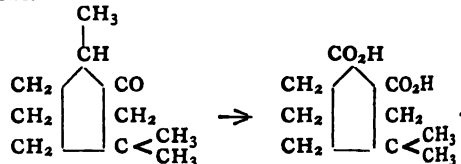
A quick and accurate method of determining albumen in milk was described by Dr. A. Trillat and E. Sauton. The sample of milk (3 c.c.) is diluted with about eight times its volume of water and boiled for five minutes. Five drops of formaldehyde are then added, and the boiling continued for a short time; then the flask is allowed to stand for five minutes, 5 c.c. of 1 per cent acetic acid added, and the whole well shaken. The precipitate is then filtered off, washed with water, freed from fat by digestion in a Soxhlet's apparatus with acetone, dried at 75° to 80°, and weighed.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

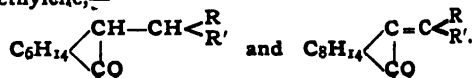
NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances de l'Académie des Sciences. Vol. cxlii., No. 18, April 30, 1906.

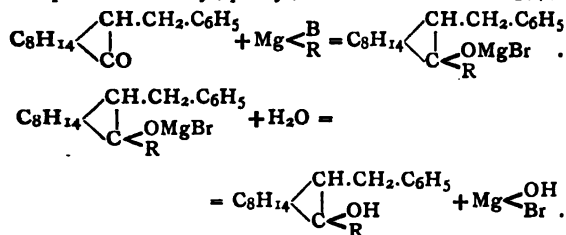
Synthesis of Pimelic $\beta\beta$ -Dimethyl and $\beta\beta$ -Trimethyl.—G. Blanc.— $\beta\beta$ -Dimethylpimelic acid is of the same importance amongst the terpenic compounds as that of $\alpha\alpha$ - and $\beta\beta$ -dimethylglutaric and adipic acids. It is, in fact, the product of degradation nearest to tetrahydrocarvone—a fact which shows the constitution of this ketone.



Diphenyl or Alcoylphenyl Camphomethane and Methylene,—



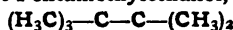
—A Haller and E. Bauer.—The preparation of secondary and tertiary benzylborneols and of the benzylcamphenes, their products of dehydration, led the authors to attempt also the preparation of dibenzyl, phenylbenzyl, and the tertiary alcoylbenzyl borneols. These derivatives are prepared by treating benzylcamphor with organo-magnesium compounds of benzyl, phenyl, and the radicals $C_n H_{2n+1}$.



After preparation, the authors then investigate the properties of the various compounds.

No. 19, May 7, 1906.

Synthesis of Pentamethylethanol,—



—Louis Henry.—



When isobutyric ether, $(H_3C)_2-CH-CO(OC_2H_5)$, acts on magnesium-methyl bromide, $CH_3-Mg-Br$, under advantageous conditions, dimethyl-isopropyl-carbinol, $(H_3C)_2-C(OH)-CH-(CH_3)_2$, is produced. In the same manner, by using ethyl chloroisobutyrate, $(CH_3)_2-CCl-CO(OC_2H_5)$, the chlorohydrine of this alcohol, $(H_3C)_2-CCl-C(OH)-(CH_3)_2$, can be obtained; and from this, under favourable conditions, the author obtains the pentamethylethanol, $(H_3C)_3-C-C(OH)-(CH_3)_2$, which, on oxidation, gives trimethylacetic acid, $(H_3C)_2-CO(OH)$.

Action of Ammonia Gas on Anhydrous Neodymium Chloride.—C. Matignon and R. Tranroy.—At a temperature of 1000° ammonia gas has no action on neodymium chloride. At ordinary temperatures, however, the anhydrous chloride absorbs dry ammonia gas, and during the process increases considerably in volume and acquires a rose tint. A considerable amount of heat is evolved. The authors investigate the compounds thus formed, and show the existence of the following addition compounds:— $NdCl_3 \cdot NH_3$, $NdCl_3 \cdot 2NH_3$, $NdCl_3 \cdot 4NH_3$, $NdCl_3 \cdot 5NH_3$, $NdCl_3 \cdot 8NH_3$, $NdCl_3 \cdot 11NH_3$, $NdCl_3 \cdot 12NH_3$. They also calculate that a total of $147\frac{1}{2}$ calories are evolved when the ammonia gas unites with one molecule of the chloride.

Existence of Phosphorus Sulphides. Mixtures of Phosphorus and Phosphorus Sesquisulphide.—R. Boulouch.—The author proves the existence of a eutectic in which the concentration of sulphur is 0.335 and the fusion-point exactly -40° . A mixture having the same composition gradually solidifies from $+35^\circ$, and the solid resulting does not constitute a eutectic. The point of eutecty of the mixtures examined is about -7° , and corresponds to a mixture of concentration of 0.200 (P_2S , instead of P_4S_3). The error the author attributes to two causes, which he carefully explains.

Brass.—Léon Guillet.—Industrial brasses which contain more than 55 per cent of copper, and until 63 per cent of copper is present, can be hammered when hot; and when the amount of copper exceeds 60 per cent, they can be hammered and beaten out in the cold. The author investigates certain brasses containing a small percentage of such bodies as aluminium, manganese, silicon, &c., and finds that the presence of foreign elements creates a fictitious standard. The presence of a constituent, in combination or solution, other than that which is recognised in the copper-zinc alloys, considerably diminishes the mechanical value of the alloy.

Estimation of Small Quantities of Iron.—A. Mouneyrat.—If a current of sulphuretted hydrogen passes through a dilute alkaline solution of an iron salt, this solution, independently of the precipitate of sulphide which is formed, turns a deep green. An investigation of this coloration led the author to discover an extremely sensitive reaction for iron, much more sensitive than the sulphocyanide reaction. It is especially useful for the detection of this element in aqueous solutions containing mere traces. The intensity of the green colour of solutions containing between $1/1,000,000$ and $1/1000$ part of iron is proportional to the amount of iron present.

Preparation of Aromatic Sulphamates by Reduction of their Nitrated Derivatives with Sodium Hydrosulphite.—A. Seyewetz and M. Bloch.—The aromatic sulphamates of general formula $(R)N.H.SO_3(M)$ have been obtained by various methods. Either a simple or substituted amine or a hydroxylamine are used in the NH_2 or $NHOH$ group, of which the sulphonic group is substituted by means of sulphuric chlorhydrin, amido-sulphuric acid, sulphurous acid, or thionylamine. The authors' method consists in directly transforming the NO_2 group into $NH-SO_3Na$, and thus obtaining the corresponding sulphamate with good yields, owing to the reduction of the nitrated derivatives with sodium hydrosulphite.

MISCELLANEOUS.

Society of Public Analysts.—Owing to the fact that the Anniversary Dinner of the Society takes place on June 13th, the ordinary meeting announced for Wednesday, the 6th inst., was postponed to Thursday, the 14th inst.

Literary Intelligence.—At a time when so much interest is centreing around the disclosures at the slaughter-houses of Chicago, the appearance of an authoritative work bearing on the subject is opportune. Messrs. J. and A. Churchill are just about to publish "Preservatives in Food and Food Examination," by Dr. John C. Thresh, Lecturer on Public Health at the London Hospital Medical College, and Medical Officer of Health for Essex, and Dr. Arthur E. Porter, Assistant Medical Officer of Health and Chief Sanitary Inspector, City of Leeds. In this work of 500 pages a critical examination is made of the various preservatives used in beverages and foods, while the question of unsound food is fully discussed. The authors maintain that "during recent years it has become notorious that serious and widespread epidemics of illness are attributable to the use of food which has become infested with organisms capable of producing disease." The text is elucidated by well executed plates.

Oxidation of Nitrous Acid by Hydrogen Peroxide. **Determination of Nitrate in presence of Nitrite.**—M. Busch.—Hydrogen peroxide is a convenient reagent to oxidise nitrous acid quantitatively to nitric acid. The reaction, $HNO_2 + H_2O_2 = HNO_3 + H_2O$, occurs very rapidly in acid solution at a temperature of 60° to 70° . If to a liquid containing a nitrite excess of neutral hydrogen peroxide solution is added, the whole warmed to 70° , and acidified with very dilute sulphuric acid, no nitrous fumes escape, and no trace of nitrous acid remains when the reaction is completed. When both acids are present the HNO_3 is best estimated gravimetrically by means of nitron acetate solution. In one half of the solution the nitrous acid is determined volumetrically by means of permanganate and the other half is oxidised with hydrogen peroxide, and then the total quantity of the two acids is weighed as nitron nitrate. The difference gives the nitric acid present. The simplest way is to precipitate with nitron acetate the liquid obtained after the titration with potassium permanganate. (Nitron = diphenyl-endanilo-dihydrotriazole; see article by M. Busch on "Gravimetric Estimation of Nitric Acid," *Berichte*, 1905, xxxviii., 861,

abstracted in CHEMICAL NEWS, 1905, xci., 234).—*Berichte*, xxxix., No. 6.

Hexamethylethane: a New Octane,

$(\text{H}_3\text{C})_3\text{—C—C—}(\text{CH}_3)_3$.—Louis Henry.—This interesting hydrocarbon is formed as a by-product in the synthetic preparation of pinacolic alcohol, $(\text{H}_3\text{C})_3\text{—C—CH(OH)—CH}_3$, when acetic aldehyde, $\text{H}_3\text{C—CHO}$, acts on a mixture of magnesium with tertiary butyl bromide, $(\text{H}_3\text{C})_3\text{—CBr}$. The hexamethylethane formed is a crystalline white solid with penetrating odour. It volatilises and rapidly disappears in free air. It melts at $103\text{—}104^\circ$ in a closed capillary tube, and boils at $106\text{—}107^\circ$ under 765 m.m. pressure. Its vapour density is 3.93. This hydrocarbon completes the series of the double symmetric methylation of ethane.—*Comptes Rendus*, cxlii., No. 20.

NOTES AND QUERIES.

"Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns."

Amorphous Elements and Liquid Air.—Will any reader kindly give particulars where I may obtain information as to any marked change in the amorphous forms of the non-metallic elements when subjected to intense cold, as in that of liquid air. I am under the impression that the waxy variety of phosphorus is converted into the red variety, exceedingly liable to most violent explosion. Is there a corresponding alteration in specific gravity? Does the crystalline variety of phosphorus undergo any marked change under similar conditions?—G. W. C.

MEETINGS FOR THE WEEK.

MONDAY, 11th.—Royal Institution, 5. General Monthly Meeting. Society of Chemical Industry, 8. "Recent Progress in the Cement Industry," by Bertram Blount. "Purifying and Stabilising Guncotton," by R. Robertson.

THURSDAY, 14th.—Society of Public Analysts, 8. (Postponed from the 6th inst.).

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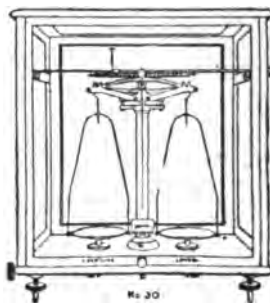
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VOL. XCIII., No. 2429.

ON THE ATTACK OF PLATINUM BY SULPHURIC ACID.*

By M. QUENNESSEN.

THE action of sulphuric acid on platinum has given rise to many memoirs, among the most important of which we may mention those of Scheurer-Kestner (*Comptes Rendus*, 1878, lxxxvi., 1082; 1880, xci., 59; *Bull. Soc. Chim.*, 1875, [2], xiv., 501; 1878, xxx., 28) and Conroy (*Journ. Soc. Chem. Ind.*, 1903, xxii., 465). The first states that the presence of nitrous products hastens the solution of platinum in sulphuric acid; the second, on the contrary, attributes to them a retarding action.

Quite recently M. Delépine (*Comptes Rendus*, 1905, cli., 866, 1013; *CHEMICAL NEWS*, 1906, xciii., 108) has recorded a much stronger attack of the metal than is shown by the figures given by Scheurer-Kestner; moreover, according to his observations the addition of nitric acid to the sulphuric acid has no influence on the rapidity of the attack.

The question did not appear to me to be nearer solution than it was before, and I therefore undertook with *pure* sulphuric acid a few researches which have thrown new light on this problem, so important in the large chemical industries.

After having ascertained by the ferrous sulphate and the diphenylamine methods that the sulphuric acid employed was quite free from nitrous compounds, I first studied the action of acid containing 94 per cent SO_4H_2 on commercial platinum. With this object I took metal serving for the manufacture of the vessels used for concentrating this acid, and I purposely exaggerated the temperature by going far beyond the maximum of 280° attained in industry for obtaining acid of 94 per cent monohydrate. Finally, I repeated the same experiments with *pure* platinum specially prepared for this purpose. Also, to avoid any action of the acid sulphate of potassium employed by M. Delépine to raise the point of ebullition, I operated in a sealed tube between 390° and 400° . Sainte-Claire Deville, moreover, showed that this salt attacks platinum at the level of the air zone.

The results of these experiments, all of which had a duration of nine hours at 400° , are given by reducing the attack to a square decimetre of metal during one hour (50 c.m.² on each side).

With commercial platinum the heating was effected—(1) in a vacuum, and (2) in an atmosphere of oxygen.

In a vacuum the metal dissolved was 0.001 grm. per decimetre-hour, and in oxygen it was 0.124 grm. In the second experiment, on opening the tube I found there was a partial vacuum, corresponding to 35 m.m. of mercury for the volume of the tube employed. There therefore was an absorption of oxygen.

With *pure* platinum in the same conditions the results were as follows:—

In the tube filled with oxygen the attack was not more than 0.0227 grm. per decimetre-hour, and in that *in vacuo* it was 0.0006 grm.

In these four experiments the sheets of metal were rolled in a spiral form; I therefore was able to see that only those parts immersed in the acid were attacked, the surfaces out of the liquid having preserved their brilliancy.

Commercial platinum is strongly etched (*moiré*), while the pure metal is only brightened.

Having established these facts I have continued the

experiments exclusively with *pure* platinum, in presence of acid of different degrees of concentration and only in a vacuum, the intervention of oxygen having been clearly established by the first experiments.

With acid, SO_4H_2 , containing about 2 per cent excess of anhydride, the attack was 0.0265 grm. per decimetre-hour, practically the same as that of pure metal in oxygen, and on opening the tube I observed the decided presence of sulphurous acid, which I also noticed in the following two experiments.

With an acid of 98.6 per cent of SO_4H_2 the attack was 0.0076 grm. per decimetre-hour, while with acid of 96.75 per cent the loss was only 0.0014 grm. In this latter experiment the temperature accidentally rose to 450° for one hour.

To sum up, I conclude that in the first instance, with an acid such as is prepared for commercial use, it is the atmospheric oxygen which acts as an oxidising agent, while with sulphuric acid of greater strength, in the absence of free oxygen, it is the sulphuric anhydride in solution in sulphuric acid which furnishes the oxygen necessary for oxidation, in accordance with a phenomenon of the same character as that which serves for the preparation of sulphurous acids with the common metals. The mechanism will be understood by reference to the work of Dittmar (*Journ. Chem. Soc.*, 1869, [2], vii., p. 446) on the dissociation of SO_4H_2 into $\text{SO}_3 + \text{H}_2\text{O}$ in sulphuric acids of unusual strength.

INVESTIGATIONS ON THE OCCURRENCE OF CHANGES IN THE TOTAL WEIGHT OF SUBSTANCES UNDERGOING CHEMICAL TRANSPOSITIONS.*

(SECOND COMMUNICATION).

By H. LANDOLT.

IN the year 1893 I published in the *Sitzungsber.*, pp. 301—334, an experimental article on the question whether in chemical transpositions the total weight of the substances taking part in the reaction remains absolutely unaltered, or whether small variations can be detected. We can conceive of the possibility of the latter alternative if we suppose either that gravity does not act with exactly the same intensity upon different substances, or that the total mass suffers an increase or decrease. The latter supposition could be explained by an hypothesis put forward by Lothar Meyer, according to which, besides the particles of the material, the ether also enters into the composition of the chemical atom, and is perhaps not absolutely imponderable; the amount of it may possibly alter during the reaction, and it may be able to pass through the walls of the vessel.

The experiments extended to reactions which occur in aqueous solution. The substances were introduced separately into the two limbs of a glass vessel shaped thus, [1]; the open ends of the vessel were then fused up, two such pieces of apparatus (A and B) of almost equal weight and of the same external volume being prepared and placed upon the two scale pans of the balance. The difference of weight of these two vessels was then determined—(1) in the original condition, (2) after the reaction had taken place in apparatus A, (3) after the reaction had taken place in B, by which each experiment was performed twice.

The investigation included the following transpositions, the substance named second being always present in excess:—

1. $\text{Ag}_2\text{SO}_4 + 2\text{FeSO}_4 = 2\text{Ag} + \text{Fe}_2(\text{SO}_4)_3$.
2. $\text{HIO}_3 + 5\text{HI} = 6\text{I} + 3\text{H}_2\text{O}$.
3. $2\text{I} + 2\text{Na}_2\text{SO}_3 = 2\text{NaI} + \text{Na}_2\text{S}_2\text{O}_6$.
4. $\text{C}_2\text{Cl}_3\text{H}_3\text{O}_2 + \text{KOH} = \text{CHCl}_3 + \text{CHKO}_2 + \text{H}_2\text{O}$.
5. $\text{C}_2\text{Cl}_3\text{H}_3\text{O}_2 + \text{water (solution process)}$.

* Communicated by the Author, being a Paper presented to the Académie des Sciences.

* From *Sitzungsber. d. K. Akademie d. Wissensch.*, 1906, viii., 266.

In Table I., Col. I. contains the details of the quantities of the substances taking part in the reactions; Col. IV. the observed alteration in weight in m.grms.; Col. V. the mean error affecting the weighing.*

I.		II.		III.	IV.	V.
Mass of Reagents.		No.	Vessel.		Observed alteration in weight.	Error in weighing.
					M.grms.	M.grms.
REACTION I.—Silver Sulphate and Ferrous Sulphate.						
Silver separated	30	1	A		-0.0167	±0.0021
		2	B		-0.0131	0.0030
	60	3	B		-0.0130	0.0017
REACTION II.—Iodic Acid and Hydriodic Acid.						
Iodine separated	64.9	4	A		-0.0047	±0.0022
		5	B		-0.0114	0.0013
		6	A		-0.0103	0.0022
	80.0	7	B		-0.0102	0.0016
		8	A		-0.0177	0.0012
	160.0	9	B		-0.0011	0.0013
REACTION III.—Iodine and Sodium Sulphite.						
Iodine used up	90	10	A		+0.0105	±0.0008
		11	B		-0.0031	0.0017
		12	A		+0.0002	0.0020
	110	13	B		-0.0127	0.0017
REACTION IV.—Chloral Hydrate and Potassium Hydroxide.						
Chloral hydrate used	150	14	A		+0.0012	±0.0024
		15	B		+0.0007	—
REACTION V.—Chloral Hydrate and Water.						
Chloral hydrate	312	16	A		-0.0003	±0.0013
Water	104					

As may be seen from Table I., in the first two reactions alterations in weight occurred, in amounts which mostly greatly exceeded the errors in weighing. But as it had appeared that, firstly, of the six experiments with iodic and hydriodic acids two (Nos. 4 and 9) gave hardly any change, and, secondly, in the reaction between iodine and sodium sulphite (Nos. 10 to 13) both positive and negative numbers occurred, and finally no definite proportionality between the reaction mass and the alteration of weight could be detected, it still seemed conceivable that experimental errors of an external kind had prevented perfect constancy of weight. I therefore stated that the variations had not been definitely proved, but at the end of the paper I added that it would be interesting to examine the repeated decreases of weight which had been found when silver and iodine were reduced to see if they always occurred in a series of further experiments, because at any rate it was not perfectly certain that they were all due to errors of observation.

As regards the above experimental results, R. v. Lieben (*Physikalische Zeitschrift von Riecke und Simon.*, 1900, I., 237) suggested that they might possibly be connected with the alterations in the dissociation proportions occurring in the reactions, or to the appearance or disappearance of electrons. After it had already been shown in another way (cathode rays) that they have a definite mass, it was clear that decreases of weight must take place in the transposition II. mentioned in the table, as well as in I., where a repression of the total dissociation occurs, and, on the contrary, increases in III., as was observed in Experiment 10. The processes IV. and V., on account of the exceedingly small dissociation, would lead us to expect no alteration in weight, which agrees with the observations. With regard to this hypothesis, it may be remarked that the experiments subsequently performed by Heydweiller

and by me, which gave a decrease instead of the expected increase when salts dissolved, are not in harmony with it.

Experiments of Other Observers.

As was mentioned in the first paper, in 1891 Kreichgauer, of Berlin, investigated whether, in the chemical union of mercury with bromine or iodine in a closed vessel, the total weight is altered, and he thus obtained only very small differences. After the appearance of my paper the subject was at once taken up by a number of other observers, among whom are the following:—

(a) F. Sanford and L. E. Ray (*Physical Review*, 1897 v., 247) examined the reaction between ammoniacal solution of silver nitrate and grape-sugar, using the method applied by me, but not weighing so accurately. When about 60 grms. of silver were reduced five experiments gave the numbers:—

Expt. No.	Observed alteration in weight.	Probable error in weighing.
	M.grm.	M.grm.
1.	-0.05	±0.07
2.	-0.05	±0.05
3.	-0.03	±0.07
4.	+0.04	±0.04
5.	+0.08	±0.04

Thus, both positive and negative differences appear and of the same order of magnitude as the errors in weighing.

(b) In 1901, A. Heydweiller (*Drude's Ann. d. Physik.*, 1901, v., 394; [Prelimin. Comm. in the *Physikal. Zeitschrift*, 1900, I., 527] published an exhaustive article on alterations in weight during chemical and physical changes. The experiments were performed with two Π -tubes, and every precaution was taken. A difference from my method lay in the fact that the vessels were not made of equal volume by the addition of substances, but their difference of volume was determined, and the unequal upward pressure in weighing was corrected by determining the density of the air. The weight of an apparatus when prepared amounted to about 300 grms., and that of the substances used + water to about 200 grms. The probable error of the mean value of the weighings, according to Heydweiller, amounted to ± 0.01 m.grm., and he assumed that alterations in weight, which exceeded 0.04 m.grm., could not be ascribed to experimental errors.

The experiments given in Table II. were performed. As appears at once from the table there is a great preponderance of negative signs, and thus the results resemble mine, which also gave decreases for the most part. As regards the separate processes we notice:—

I. The reaction between Fe and CuSO_4 proceeded without appreciable alteration of weight, if the copper vitriol used was free from acid (Expts. 1 and 2); on the other hand, a decrease far exceeding the experimental error (0.04 m.grm.) occurred if the solution contained only a very small quantity of alkali (Expts. 3, 4, and 5), or sulphuric acid (Expts. 6 and 7). The action of these substances is obscure.

II. On dissolving copper vitriol free from acid in water again hardly any decrease occurred (Expt. 8), but there was a very large decrease when ordinary salt was used (Expt. 9), or when sulphuric acid was added (Expts. 10 and 11).

III. On mixing copper sulphate solution with dilute sulphuric acid no alteration of weight took place (Expt. 12).

IV. The decomposition of copper sulphate with caustic potash (Expts. 13 to 18) was always accompanied by a decrease of weight, which, on partial mixing of the liquids, is smaller than on complete mixing (Expts. 13, 14, and 16, 17).

V. The small alterations of weight which appeared on neutralising acetic acid with ammonia (Expts. 19 and 20) are within the limits of experimental error (0.04 m.grm.).

VI. The same holds good when barium chloride is decomposed by sulphuric acid.

* In the former paper the error given was the probable and not the mean, the first of which amounts to two-thirds of the second.

TABLE II.

Expt. No.	Contents of the two limbs of the vessels.	Observed alteration in weight.
I. Iron and Copper Sulphate.		
a. Solution Neutral.		
1.	13.96 grms. Fe; 63.8 grms. $\text{CuSO}_4 \cdot 5 \text{ aq}$ + 100 grms. water	M. grms. - 0.026
2.	13.96 grms. Fe; 63.1 grms. $\text{CuSO}_4 \cdot 5 \text{ aq}$ + 100 grms. water	+ 0.019
b. Solution Alkaline.		
3.	15 grms. Fe; 79.9 grms. $\text{CuSO}_4 \cdot 5 \text{ aq}$ + 130 grms. water with trace of NaOH	- 0.217
4.	15 grms. Fe; 69.6 grms. $\text{CuSO}_4 \cdot 5 \text{ aq}$ + 115 grms. water with 0.13 gm. NaOH	- 0.161
5.	18.3 grms. Fe; 98.0 grms. $\text{CuSO}_4 \cdot 5 \text{ aq}$ + 103 grms. water with 0.23 gm. NaOH	- 0.176
c. Solution Acid.		
6.	15 grms. Fe; 69.6 grms. $\text{CuSO}_4 \cdot 5 \text{ aq}$ + 114.2 grms. water with 0.36 gm. H_2SO_4	- 0.097
7.	18.3 grms. Fe; 103.2 grms. $\text{CuSO}_4 \cdot 5 \text{ aq}$ + 92 grms. water with 0.06 gm. H_2SO_4	- 0.158
II. Solution of Copper Vitriol in Water.		
8.	62 grms. salt crystallised from alkaline solution; 151 grms. water	- 0.029
9.	62 grms. ordinary copper vitriol; 147 grms. water	- 0.126
10.	50 grms. ordinary salt; 150 grms. water containing 7.3 grms. H_2SO_4	- 0.081
11.	50 grms. ordinary salt; 150 grms. water containing 3.7 grms. H_2SO_4	- 0.072
III. Mixture of Copper Sulphate Solution with Dilute Sulphuric Acid.		
12.	38 grms. $\text{CuSO}_4 \cdot 5 \text{ aq}$ + 110 grms. water; 2.37 grms. H_2SO_4 + 10 grms. water ..	+ 0.014
IV. Mixture of Copper Sulphate Solution with Caustic Potash.		
(38 grms. $\text{CuSO}_4 \cdot 5 \text{ aq}$ + 110 grms. water; 2.25 grms. KOH + 10 grms. water).		
13.	After addition of half the caustic potash ..	- 0.037
14.	After addition of all the caustic potash ..	- 0.092
(33 grms. $\text{CuSO}_4 \cdot 5 \text{ aq}$ + 92 grms. water; 10.04 grms. KOH + 40 grms. water).		
15.	After complete mixing in Vessel A	- 0.068
16.	After half mixing in Vessel B	- 0.059
17.	After complete mixing in Vessel B	- 0.080
18.	34.4 grms. $\text{CuSO}_4 \cdot 5 \text{ aq}$ + 99.5 grms. water; 13.4 grms. KOH + 15 grms. water	- 0.045
V. Acetic Acid and Ammonia.		
19.	49.7 grms. $\text{C}_2\text{H}_4\text{O}_2$ + 87.5 grms. water; 15.3 grms. NH_3 + 123.7 grms. water ..	- 0.034
20.	50.4 grms. $\text{C}_2\text{H}_4\text{O}_2$ + 88.5 grms. water; 15.6 grms. NH_3 + 125.9 grms. water ..	- 0.026
VI. Barium Chloride and Sulphuric Acid.		
21.	20.0 grms. BaCl_2 + 100 grms. water; 9.7 grms. H_2SO_4 + 40.3 grms. water ..	- 0.016

As Heydweiller emphasises, no connection can be observed between the alterations in weight, and the other physical and chemical processes occurring during the reaction, they appear both with increases (Group II.) and decreases (IV. and VI.) of the electrolytic dissociation, density (II. and VI.), and magnetic permeability (I.).

With regard to Heydweiller's experiments, Lord Rayleigh (*Nature*, 1901, lxiv., 181) remarked that there was not

always a state of equilibrium in the vessels before the transposition, as, for example, in Group II., when solid copper vitriol was in one limb and water in the other. By continuous distillation of the water, changes of temperature might be caused, and they would possibly influence the determination of the weights. In a rejoinder of Heydweiller's (*Physikal. Zeitschrift*, 1902, iii., 425) he points out that if this were the cause of the decreases of weight observed in Experiments 9 to 11, the action would also have manifested itself in Experiment 8, where, however, practically no alteration appeared.

(c) In 1903, J. Joly ("On the Conservation of Mass," *Royal Dublin Soc. Trans.*, 1903, Ser. 2, viii., 23-52), of Dublin, in quite a different way, investigated whether, on dissolving copper vitriol in water, any change of mass could be observed. Stated shortly, the method consisted in hanging on one end of a torsion balance a glass vessel containing the two substances at first separated, and when at mid-day or midnight the arms were perpendicular to the direction of the earth's motion, the salt was allowed to dissolve. Acceleration would be obliged to occur if mass disappeared, and conversely. Of fourteen observations eight argued definitely and three less clearly for a decrease of mass, two were against it, and the last was doubtful.

(d) In 1903, G. Kahlbaum performed experiments on the question whether, when the grey modification of tin is converted into the white, and *vice versa*, any change of weight can be observed. The weighings showed no such change.

(e) In 1904, A. Lo Surdo (*Nuovo Cimento*, 1904, Ser. 5, viii.), of Messina, investigated very carefully the reaction between iron and copper sulphate. He used Ω -shaped vessels of Thuringian glass, which, as in Heydweiller's experiments, contained on the one side about 15 grms. of iron powder, and on the other about 80 grms. of copper sulphate and 200 to 250 grms. of water to which a small quantity of caustic soda had been added. The external volume of the apparatus, adjusted to 0.004 to 0.023 c.c. by addition bodies, was determined before and after the reaction, when the alterations denoted below by *b* were observed. The weighings were performed with a balance fitted with mirror reading arrangement from Sartorius, of Göttingen (sensitiveness, 20 to 30 scale divisions per m.grm.). The balance possessed an arrangement by which the vessels could not only be exchanged but also inclined, so that the reaction could be carried out within the balance case without touching the glass. The probable error of the mean of six or seven single weighings amounted to ± 0.003 m.grm. to ± 0.007 m.grm., and in one case to ± 0.012 m.grm.

Five weighings gave the following results:—

Expt. No.	a. Change of weight. M.grm.	b. Change of volume. C.c.
1.	+ 0.008	+ 0.011
2.	- 0.008	+ 0.002
3.	- 0.008	+ 0.008
4.	+ 0.013	+ 0.003
5.	+ 0.003	- 0.006

As the experimental errors are estimated at 0.02 m.grm. at the most the alterations of weight lie completely within this limit, and they would not differ appreciably if corrections for change of volume were applied. Hence, Lo Surdo concluded that during chemical reactions no perceptible differences of the total weight can be observed.

But it must be noted that this statement applies only to the reaction between iron and copper sulphate.

In view of the contradictory results obtained by the various workers, a fresh investigation of the problem was an urgent necessity. It had to be decided definitely whether the decreases of weight found repeatedly by Heydweiller and myself for several reactions were caused only by experimental errors, produced by purely external causes of little interest, or whether they were connected with the alteration of the substances.

As according to previous experience* the effect of any inequality of gravity upon different bodies is infinitely small, from the axiom of the conservation of matter, the cause of the observed decreases of weight can be sought only in a decrease of mass. In my first paper I took into consideration the presence of ether, either contained in the atoms (Lothar Meyer) or strongly condensed round them (C. Nägeli), and partially escaping during the reaction. Also Lieben's view, that the alterations in weight were possibly connected with the appearance or disappearance of electrons, was mentioned. Moreover, in the present age of radio-activity, when the spontaneous alterability of the atoms of several elements has been recognised, the conjecture may be expressed, that in consequence of the violent shock which the atoms suffer in chemical reactions, both radio-active and other elements may have a small part of their mass split off. If it is possible that the released particles on account of their minuteness can pass through the walls of the vessel, in chemical transpositions decreases of weight would appear, but no increases.

When I resolved to take up the subject again, the earlier experiences, acquired in the years 1890-1892, led me to expect a very lengthy and tedious piece of work. It had been found that the alterations of weight which occurred never exceeded 0.17 m.grm., and mostly amounted to hundredths of a m.grm., a region in which the weighing of glass vessels might be affected by many sources of error. The only hope of obtaining more certain results lay in considerably increasing the accuracy of the method, and especially of the weighing. Luckily this was made possible owing to the exceedingly acceptable assistance of the Akademie der Wissenschaften and of the Kgl. Cultusministerium, through whom I obtained possession of a new and excellent balance, besides other necessary instruments. A beginning was made with the experiments at the end of the year 1901; they gave almost always decreases of weight in different reactions, but there was still often a doubt whether they were not to be ascribed to external influences. I could not feel confidence in the results till an extended series of experiments had been finished in which the vessels were filled with substances not capable of reacting with one another, and then treated in exactly the same way as in the experiments with substances which underwent chemical transposition. Under these circumstances decreases and increases of weight occurred in almost equal numbers, and were, moreover, very considerably smaller than those occurring during the reactions. As will be explained in full later, the greatest error which affected the whole method, including the weighing, was proved to be ± 0.03 m.grm.

In this communication I must limit myself to a statement of the results and a short description of the methods. The material used for the observations was very extensive, and I have therefore been obliged to prepare another complete report of the whole work, which I shall present for the *Transactions of the Akademie*.

(To be continued).

Royal Institution.—A General Monthly Meeting of the Members of the Royal Institution was held on the 11th instant, the Rt. Hon. Sir James Stirling, Vice-President, in the Chair. Mrs. Elgar, Miss Hilda Hanbury, Mr. E. L. Mansergh, Mrs. Morse, and Mr. C. D. Page were elected Members. The Special Thanks of the Members were returned to Sir Andrew Noble, K.C.B., F.R.S., for his donation of £100, and to Mrs. Frank Lawson for her donation of £50 to the Fund for the Promotion of Experimental Research at Low Temperatures.

* According to the observations of R. von Eötvös and D. Kreichgauer mentioned in the first paper, it is to be assumed that if, generally speaking, there is a difference in the weight of bodies of the same mass, but of different material, this amounts to less than a twenty millionth part of the measured magnitude. For a reacting mass of 100 to 200 grms. as was used in the experiments here referred to, the difference would amount to 0.005 to 0.010 m.grm., and would thus lie within the limits of experimental error.

THE BEARING OF HYDRATES ON THE TEMPERATURE COEFFICIENTS OF CONDUCTIVITY OF AQUEOUS SOLUTIONS.

By HARRY C. JONES.

THAT the electrical conductivity of aqueous solutions of electrolytes, in general, increases greatly with rise in temperature is a well known fact. This might be due either to an increase in the dissociation of the electrolyte with rise in temperature, or to an increase in the velocity with which the ions move, or to both. It is not a difficult matter to test the effect of change in temperature on the dissociation of electrolytes. It is only necessary to measure the dissociation directly, at different temperatures, by the conductivity method. This has been done recently by Jones and West (*Am. Chem. Journ.*, 1905, xxxiv., 357), for temperatures ranging from 0° to 35°. The result is that electrolytes, in general, are *slightly less* dissociated at the higher than at the lower temperatures.

As has been pointed out, this is in accord with the theory of Dutoit and Aston, which makes the dissociating power of a solvent a function of its own association—the more associated a solvent the greater its dissociating power. Take a solvent like water, the higher the temperature the less it is associated, and, consequently, the smaller its power to break molecules of electrolytes down into ions.

Having eliminated the factor of dissociation as increasing the conductivity of electrolytes at the higher temperature, we are forced to conclude that the increase in conductivity with rise in temperature, shown by solutions of electrolytes in general, is due to an increase in the velocities with which the ions move.

There are a number of factors which determine the velocity with which an ion moves through a solution of an electrolyte. Assuming that the force which drives the ions is constant, the velocity would be conditioned chiefly by the viscosity of the medium through which the ion passed, and by the size of the ion. At the more elevated temperature the force which drives the ion would be greater, and the viscosity of the medium through which the ion moves would be less. Both of these factors would increase the velocity with which the ions move, and, consequently, increase the conductivity as the temperature was raised.

The object of this paper is to call attention to another factor which causes the ions to move faster at the more elevated temperature. The mass of the ion decreases with rise in temperature. This does not refer to the charged atom or group of atoms which we usually term the ion, but to this charged nucleus plus a larger or smaller number of molecules of water which are attached to it, and which it must drag along with it in its motion through the remainder of the solvent.

That ions are hydrated has been shown beyond question by Jones and his co-workers. That these hydrates are relatively unstable compounds has also been demonstrated; the higher the temperature the less complex the hydrates existing in the solution. This can be seen from one example. In a solution of a certain definite concentration, every molecule of calcium chloride or the ions resulting from it holds about 30 molecules of water. From such a solution practically all of the water can be removed by simply boiling it, except 6 molecules of water to 1 of calcium chloride—this number being brought out of the solution by the salt as water of crystallisation. The higher the temperature, then, the less complex the hydrate formed by the ion. The less the number of molecules of water combined with the ion the smaller the mass of the ion, and the less its resistance when moving through the solvent. Consequently, the ion will move faster at the high temperature. This conclusion can be tested by the results of experiment. If this factor of diminishing complexity of the hydrate formed by the ion with rise in temperature, plays any prominent rôle in determining the large tem-

perature coefficient of conductivity, then we should expect to find those ions with the largest hydrating power having the largest temperature coefficients of conductivity. This will readily be seen to be the case. The more complex the hydrate, i.e., the greater the number of molecules of water combined with an ion, the greater the change in the complexity of the hydrate with rise in temperature. We can readily test this conclusion by the results of the experimental work of Jones and West (*Am. Chem. Journ.*, 1905, xxxiv., 357). Let us compare the temperature coefficients of conductivity per degree rise in temperature, for some of those substances which have slight hydrating power, with the corresponding coefficients for a few of the substances which have a much greater power to combine with water.

The volumes for which the comparisons are made range from 2 to 1024, and the temperatures from 25° to 35°.

A comparison of Table I. and Table II. will show that the above conclusion is confirmed by the experimental results. The substances included in Table I. have very slight hydrating power. Those in Table II. have very much greater hydrating power. It will be remembered that hydrating power is a function of water of crystallisation—the larger the number of molecules of water of crystallisation the greater, in general, is the hydrating power of the substance. It will be seen that the substances in Table I. have little or no water of crystallisation, while those in Table II. crystallise with large amounts of water. The water of crystallisation may be taken as roughly proportional to the hydrating power of the substance.

The substances in Table I. have much smaller coefficients of conductivity than those in Table II., even taking into account the fact that those in Table II. are ternary electrolytes, while those in Table I. are binary electrolytes.

TABLE I.—Substances with Slight Hydrating Power.

	Temperature coefficients in conductivity units.	
	v=2.	v=1024.
Ammonium chloride ..	2.07	2.94
Ammonium bromide ..	2.16	2.86
Potassium chloride..	2.13	2.84
Potassium bromide..	2.18	2.91
Potassium iodide ..	2.09	2.91
Potassium nitrate ..	1.86	2.71

TABLE II.—Substances with Large Hydrating Power.

Calcium chloride ..	3.11	5.61
Calcium bromide ..	3.01	5.20
Strontium bromide..	2.93	5.27
Barium chloride ..	2.86	5.30
Magnesium chloride ..	2.55	4.59
Manganese chloride ..	2.37	4.86
Manganese nitrate ..	2.24	4.16
Cobalt chloride ..	2.54	4.95
Cobalt nitrate ..	2.48	4.67
Nickel chloride ..	2.63	5.04
Nickel nitrate ..	2.51	4.58
Copper chloride ..	2.15	5.04
Copper nitrate ..	2.38	4.88

Another fact, of equal importance, is brought out by comparing the results in Table I. with one another, and similarly those in Table II. with one another. If the temperature coefficient of conductivity is a function of the decrease in the complexity of the hydrate formed by the ion, with rise in temperature, then we should expect that those substances which have equal hydrating power would have approximately the same temperature coefficients of conductivity.

If we examine the above tables we shall see that this is true. The substances in Table I. all have only very slight hydrating power, as would be expected from the fact that they all crystallise without water. Their temperature coefficients of conductivity are all of the same order of magnitude, and, indeed, are very nearly equal.

The substances in Table II. all have very great hydrating power, and all have a hydrating power of the same order of magnitude. This would be expected, since nearly all of these substances crystallise with 6 molecules of water. There are a few compounds in this table calling for special comment. Barium chloride crystallises with only 2 molecules of water, yet it forms hydrates comparable with those substances with larger amounts of water of crystallisation. It is therefore perfectly in keeping with the above relation that its temperature coefficients of conductivity should be of the order of magnitude that they are in the above table.

Manganese chloride crystallises with only 4 molecules of water, but the work of Jones and Bassett (*Am. Chem. Journ.*, 1905, xxxiii., 562) shows that it forms hydrates about as complex as the other salts in Table II. Its temperature coefficients of conductivity are of the same order of magnitude as the other substances in this table.

Of the compounds recorded in Table II., the one which, apparently, presents the most pronounced exception to the relation that we are now considering, is copper chloride. This salt crystallises with only 2 molecules of water, and yet has a temperature coefficient of conductivity that is nearly as large as the salts with 6 molecules of water of crystallisation. It might be inferred that this salt has much less hydrating power than the others in Table II. The work of Jones and Bassett shows that this is not the case (*Am. Chem. Journ.*, 1905, xxxiii., 577). Copper chloride has a large hydrating power, indeed much larger than would be expected from the amount of water with which it crystallises. Its temperature coefficient of conductivity is therefore not surprisingly great.

A third point that is brought out by the results in the above tables is the following:—At the higher dilution the temperature coefficient of conductivity for any given substance is greater than at the lower dilution. That this is a general relation will be seen by reference to the work of Jones and West (*Am. Chem. Journ.*, 1905, xxxiv., 357). This is explained very satisfactorily on the basis of the suggestion made in this paper. The complexity of the hydrate at the higher dilution is greater than at the lower dilution, as is shown by the experiments of Jones and his co-workers on the composition of the hydrates formed by different substances at different dilutions (*Am. Chem. Journ.*, 1905, xxxiii., 534; 1905, xxxiv., 290).

The hydrate being more complex at the higher dilution, the change in the composition of the hydrate with change in temperature would be greater at the higher dilution, and, consequently, the temperature coefficient of conductivity is greater the more dilute the solution.

The three points that are established in this paper are:—

1. The temperature coefficients of conductivity of aqueous solutions of electrolytes are greater, the greater the hydrating power of the electrolyte.

2. The temperature coefficients of conductivity of aqueous solutions of electrolytes are of the same order of magnitude for those substances having, approximately, the same hydrating power.

3. The temperature coefficients of conductivity, for any given substance, increase with the dilution of the solution, and this increase is greatest for those substances with large hydrating power.

All three of these conclusions are necessary consequences of the assumption that the large change in conductivity with change in temperature is due, in part, to the decreasing complexity of the hydrates formed around the ions, with rise in temperature. Since these conclusions are all verified by the results of experiment, we must accept the assumption which led to them as containing a large element of truth.

It is more than probable that the decreasing complexity of the hydrates, with rise in temperature, is a very important factor in conditioning the large temperature coefficients of conductivity, especially of those substances which have large hydrating power.—*American Chemical Journal*, xxxv., No. 5.

A REVISION OF THE ATOMIC WEIGHTS OF
SODIUM AND CHLORINE.*

By THEODORE WILLIAM RICHARDS

and
ROGER CLARK WELLS.

(Continued from p. 262).

THE FUSION OF SODIC CHLORIDE IN VACUUM.

THIS matter has been given an especial heading because it is one which has never before received adequate treatment in an investigation of this kind. It is well known that many substances, when fused in the air, dissolve either oxygen or nitrogen, or both, thereby increasing sensibly their weight. While it was hardly to be expected that this would be the case with sodic chloride, the point should not pass unchallenged in a research striving to attain the highest possible accuracy.

Hence two analyses were made of sodic chloride fused for some time in a platinum boat inclosed in a completely evacuated porcelain tube, and subsequently bottled in dry air in the often-described "bottling apparatus." So far as we know, these are the only quantitative experiments ever made in which *all* the weighed substances were fused in a vacuum before weighing. It is true that Stas has shown that ammoniac chloride sublimed in a vacuum gave the same equivalent referred to the silver as preparations sublimed in air; but this choice of ammoniac chloride had the disadvantage that its two dissociation products possess different rates of diffusion, and, moreover, his silver had not been fused in a vacuum as ours had been. The data and results are given below.

The Ratio of Sodic Chloride to Silver.
(Both substances fused in a vacuum).

No.	Sample salt.	Sample Ag.	Weight NaCl. (In vac.).	Weight purest corrected Ag. (In vac.).	Parts NaCl equivalent to 100,000 parts Ag.
59.	J	P	3.38684	6.25046	54.185
60.	J	P	4.68529	8.64634	54.188

Average 54.187

These experiments show that probably not enough gas is absorbed during fusion of the salt in air sensibly to alter the apparent combining weight. It is true that gases easily remain in supersaturated solution in liquids, and that long-continued relief from pressure is necessary to remove them; but fused sodic chloride is so mobile and possesses so low a density as to make the retention of much gas very unlikely. That air may be adsorbed by the cold salt is of course highly probable, but the surface of a fused lump is so small that the weight of this adsorbed air must be negligible. One would here expect a lower result from the salt fused in a vacuum, if gas were dissolved in the other experiments, but the results were, in fact, slightly higher, although not beyond the probable limit of experimental error. It appeared from tests with indicators that the salt remained strictly neutral after fusion in a vacuum, as it did in the air.

Being unable to conceive of any other sensible inaccuracies which could affect the analyses of the chloride of sodium, we turned our attention next to the composition of the chloride of silver, because of the before-mentioned suspicion that Stas was here also in error. An additional reason for this revision is found in the fact that this atomic weight of iodine, found at about the same time, has been found to be about 0.1 per cent too low. (Ladenburg, *Ber.*, xxxv., 2275; Scott, *Proc. Chem. Soc.*, xviii., 112; Köthner and Aeuer, *Ber.*, 1904, xxxvii., 2536, and *Liebig's Ann.*, 1904, cccxxxvii., 123, 362; Baxter, *Proc. Amer. Acad.*, 1904, xl., 419).

THE SYNTHESIS OF ARGENTIC CHLORIDE.

Many accurate analyses were made by Stas which involve more or less directly the atomic weight of chlorine,

* Published by the Carnegie Institution of Washington, April, 1905.

but of these we are concerned in the present paper only with those which compare silver with chlorine, because such alone could cause an error leading to the discrepancy under discussion. The experimental solution of the problem is thus a comparatively simple matter, to be accomplished by combining silver with chlorine. Among all Stas's experiments there seems to have been only seven which dealt directly with this ratio ("Untersuchungen," trans. Aronstein, 1867, 173, 175; ex. "Oeuvres," i., 333). These were carried out by two methods. Of these methods, one, the ignition of silver in a stream of chlorine, is acknowledged by Stas to possess errors which he could not wholly correct. Hence the three experiments made by this method are of doubtful value, and only four analyses remain for the purpose of obtaining directly this very important ratio. Even in these the large glass globes employed were more or less attacked by the strong acids or the fused chloride, and, moreover, Stas questioned the purity of the silver used in one case (Aronstein, p. 117; "Oeuvres," i., 330). There is obviously no doubt of the need of obtaining more light upon this matter.

The fundamental requirements of a satisfactory synthesis of argentic chloride are as follows:—First, that the silver must be pure; secondly, that it must all be collected in the argentic chloride, and thirdly, that this argentic chloride must be pure. Because all the error accumulates upon the consequent atomic weight of chlorine and is magnified in calculation, the requirements are usually rigorous.

The fulfilling of the first of these requirements has already been treated in detail, under the discussion of the preparation of silver. Our knowledge of the essentials of this easy but subtle process grew as the quantitative work on the synthesis of the chloride progressed. Whether in the future anyone may be able to discover an unsuspected source of impurity in our purest silver, is of course impossible to say. At least there can be no doubt that it was of distinctly a higher grade than the silver used by Stas in most of his work.

In order to convert this silver wholly into the chloride without loss or illegitimate gain, two methods were used, for fear that in one of these a constant error might lurk unsuspected. The first consisted in the usual method of solution in nitric acid and precipitation in dilute solutions, being essentially similar to that used in obtaining the ratio between common salt and silver chloride. The weighed silver was dissolved with all the precautions used in previous work and precipitated as chloride by hydrochloric acid at concentrations of about fifth normal. Hydrochloric acid was added in excess and the precipitate was washed three times with dilute hydrochloric acid, in order to diminish as much as possible the solubility of the silver salt. After collection on a Gooch crucible, drying at 150°, and washing the bulk of the precipitate was fused in a porcelain or quartz crucible and the loss in weight by fusion determined.

It would appear at first as if the result of this process could furnish nothing not directly calculable from a comparison of the two sodium series, in one of which common salt was compared with silver and in the other with argentic chloride. This is not true, however, for in this previous work the precipitate formed in the presence of sodic nitrate and excess of argentic nitrate, while in the present case an occluded alkaline salt could not be carried down, because none was present, and *all* the silver could be converted into chloride. For this reason the synthesis of argentic chloride in the presence of nothing but volatile substances is an important step in the work.

As usual, in this case also, improvement was effected as the work progressed, and here as before the less perfect tentative work is included in the table (Preliminary Series). The synthesis of argentic chloride is a process of greater certainty than a metathetical reaction, for the reasons just stated, and hence, finer corrections could be discovered and applied.

These corrections, in addition to the two already

SYNTHESIS OF ARGENTIC CHLORIDE.

Preliminary Series.

No. of expt.	Description of silver. Sample, support, and environment during fusion.	Observed weight of silver (vac.).	First approximate weight AgCl (vac.).	Corrections to AgCl.				Corrected weight AgCl (vac.).	Parts of AgCl produced from 100,000 parts Ag.
				Asbestos residue (add.).	Loss on fusion (subtr.).	Gain in Cl ₂ (add.).	Cor. for solubility (add.).		
				Grms.	Grms.	M.grm.	M.grm.		
65.	P, lime, vac. . .	9.06483	12.04416	0.10	0.79	0.12	0.06	12.04365	132.861
66.		8.39217	11.14987	0.35	0.57	0.14	0.06	11.14985	132.860
70.		5.37429	7.14072	0.26	0.51	0.03	0.06	7.14056	132.865
Average									132.862
67.	P, carbon, blast	8.08222	10.73844	0.46	0.49	0.22	0.06	10.73869	132.868
68.		7.08517	9.41382	0.20	0.50	0.04	0.06	9.41362	132.864
71.		7.97715	10.59854	0.45	0.82	0.14	0.06	10.59837	132.859
Average									132.864
72.	S, lime, vac. . .	8.11978	10.78751	0.36	0.38	0.09	0.09	10.78767	132.857
73.		8.53452	11.33904	0.28	0.40	0.09	0.06	11.33907	132.861
74.		6.73284	8.94497	0.42	0.51	0.14	0.09	8.94511	132.858
75.		8.91366	11.84225	0.48	0.48	0.09	0.06	11.84240	132.857
76.		9.72295	12.91736	0.56	0.41	0.09	0.09	12.91769	132.858
Average									132.858
79.	U, carbon, H ₂ .	8.63961	11.47877	0.30	0.61	0.07	0.09	11.47862	132.860
R. 1.	V, lime, blast..	11.13795	14.79856	0.24	0.50	0.10	0.09	14.79849	132.865
Total average									132.861
Stas found									132.848

Final Series.

69.	7.24427	9.62517	0.16	0.40	0.09	0.06	9.62508	132.865
77.	8.30502	11.03312	1.10	0.14	0.67	0.09	11.03484	132.870
78.	7.29058	9.68547	1.23	0.12	0.09	0.09	9.68676	132.867
80.	8.58472	11.40578	0.57	0.32	0.02	0.09	11.40614	132.866
81.	8.01318	10.64632	0.34	0.32	0.05	0.09	10.64648	132.862
R. 2.	9.77160	12.98283	0.74	0.37	0.02	0.13	12.98335	132.868
83.	7.98170	10.60528	—	—	—	—	10.60528	132.870
Average								132.867
R. 3.	11.49983	15.27978	0.44	0.66	0.03	0.05	15.27964	132.868
84.	6.25318	8.30834	—	—	—	—	8.30834	132.866
85.	7.72479	10.26360	—	—	—	—	10.26360	132.866
Average								132.867
Total (Final Series) ..		82.66887					109.83951	132.867

heeded, namely, that for the asbestos shreds carried away during filtration and that for the inclosed wash water set free on fusion of the chloride, were five in number.

The first of these was a correction for the presence of a trace of argentic chloride clinging to the collected asbestos shreds. This silver salt lost chlorine on ignition—a quantity which was determined by dissolving the residual silver and titrating with N/100 sulphocyanate. The amount of chlorine thus lost on the average was 0.02 m.grm. in each synthesis—an amount which was added to the weight of the asbestos shreds. This correction might in most cases have been omitted, but it was determined for the sake of certainty.

The second of these corrections was for the slight solubility of argentic chloride even in dilute hydrochloric acid. This solubility may have been only apparent, the trace found being due to the imperfection of even the double filtration through asbestos and filter-paper—but in any case it needed determination and correction. The average solubility, from five determinations (p. 203), was one part in 30,000,000 of water, which is about the order of the possible solubility of undissociated argentic chloride, and

hence may have represented that quantity. The determination was made by evaporating the hydrochloric acid solutions, rinsing out the dish with ammonia, and testing the resulting solution nephelometrically by comparison with similarly prepared known solutions of silver chloride, as has been said.

The third correction was for an occasional loss of chlorine by the argentic chloride on fusion, owing either to the presence of argentic nitrate or to traces of dust. The darkening thus caused represents a real, although often trivial, deficiency of weight; but, fortunately, this is easily restored by fusion in a mixture of pure chlorine and hydrochloric gas. Of course this process must not be adopted when argentic nitrate is occluded during the analysis of a metallic chloride, for it would then lead to a large excess of argentic chloride; but it is perfectly safe in the present case. The only suspicion which attaches to it is the possibility that an excess of one of these gases might be dissolved. This is undoubtedly a real danger, unless the fused salt is exposed to the air and agitated in contact with it while fused. If this is done, we have assured our selves by repeated experiments, that no essential excess of

chlorine remains dissolved. (Dr. Baxter, *Proc. Amer. Acad.*, 1904, xl., 432, has come to the same conclusion; and so also have Köthner and Auer, *Liebig's Annalen*, 1904, cccxxxvii., 127). Two facts pointed to this conclusion:—First, that those samples of argentic chloride, which after the preliminary fusion merely in air were perfectly colourless, transparent, and pearly, gained no essential weight on fusion in chlorine (Expts. 80, R. 2, and R. 3, Final Series); and, secondly, that argentic chloride fused in chlorine, properly freed from it, and subsequently again and again fused in pure air, lost no essential weight.

We always secured elimination of the possible trace of dissolved chlorine by continuing to fuse in air with occasional removal of the cover, and by carefully rolling the inclined crucible around, while held with forceps, during the solidification of the fused mass. Spreading the solid in this way makes it easier to fuse the salt once more without breaking the very fragile quartz crucibles.

The attacking of glass by chlorine in contact with silver chloride, and a consequent loss of weight, gave Stas considerable difficulty. Baxter has found that even porcelain is attacked in displacing iodine with chlorine in the silver salts. We did not greatly fear a loss of weight of the porcelain crucibles, since our chlorine was dilute, and was applied only for a few minutes; nevertheless, we adopted the use of quartz as an added precaution in the later experiments. No difference in results could be detected by its use.

A fourth correction which we sought to determine was for the possible volatility of the argentic chloride during fusion. The constancy of weight on repeated fusion, already mentioned, seems to be sufficient proof for considering this correction as negligible. The conclusion is strengthened by the results of Biltz and Victor Meyer, who found argentic chloride to be but slightly volatile at much higher temperature (*Ber.*, 1889, xxii., 727). It is not, of course, contended that argentic chloride has no vapour-tension at 500°, but only that the loss occasioned by its evaporation is too small to receive consideration.

(Köthner and Auer, *Liebig's Annalen*, 1904, cccxxxvii., 128, found a volatilisation of only 0.0002 in a case where several litres of gas had been passed over argentic chloride during several hours. This agrees essentially with our result, for the likelihood of distillation in our case was much less).

During fusion, however, there is sometimes a slight spurring or projection upward of minute drops of fused material. Hence the crucible must always be covered with an accurately weighed lid—a precaution which was always taken in the final experiments.

The phenomenon of spurring suggested that weighable quantities of air may be absorbed during fusion in air, and led to our effort to discover a fifth correction. Carefully conducted experiments showed, however, that there was no loss in weight of 3 grms. of ordinarily fused argentic chloride when fused again in a vacuum. The fusions were conducted in a porcelain boat covered with another inverted to serve as a lid. Slight traces of enclosed gas were noticed by Stas in many substances, but our quantitative experiments showed that they do not amount to a weighable quantity either in common salt or silver chloride. Thus, all the final experiments in this paper refer to substances fused *in vacuo*, and hence presumably as free as possible from enclosed air.

There were thus seven possible corrections, besides the correction of the weights to the vacuum standard, which might be applied to the results. Two of these, that for the possible volatility of argentic chloride and that for weight of dissolved air, were so small as to be entirely negligible. The chlorine lost from the traces of argentic chloride clinging to the displaced asbestos shreds was added to the weight of these shreds, and is included therein in the table. The three other corrections are recorded in detail in the table, which contains the original weight of the precipitate dried at 150°, as well as the final corrected values. The almost negligible correction for the solubility

of argentic chloride in dilute hydrochloric acid was computed, in the Preliminary Series, from the volume of the washings.

A few inessential changes in the mode of precipitation were occasionally introduced. In Experiments 75, 76, and 79 both solutions were only about tenth normal, and the number of washings was diminished; in R. 1 the argentic nitrate was very dilute, but the hydrochloric acid was somewhat concentrated.

As has been said, the tentative experiments were all classed together in the Preliminary Series. The table explains itself, for the most part, except perhaps the second column. This defines in the first place the designation of the silver, next the nature of the containing vessel on which it was fused, and last the atmosphere surrounding the metal during fusion. The word "blast" signifies that the sample thus qualified was fused in a blast-flame of illuminating gas and air, delivered from a clean nozzle, and that the resulting mass was cooled in the reducing flame.

Although not giving the true amount of chloride to be obtained from the purest silver, this table was highly instructive. It showed in the first place that even silver of this grade of purity must be 0.01 per cent purer than that of Stas.

(There can be no doubt that this difference is due to the fact that Stas fused all the silver used in this part of his work in a porcelain crucible under borax and nitre, thereby introducing both alkali and oxygen ("Oeuvres," i., 328). His own subsequent experiments (Oeuvres," i., 456) showed that this metal was at least 0.005 per cent less pure than his distilled granulated silver, which must, in its turn, have contained some oxygen. His still later elaborate search for oxygen in the silver is not satisfactory, because just before determining the oxygen in it he ignited it in a reducing flame, and because he sought to determine the oxygen only by heating to redness in a vacuum, a process which may have set free only the superficial oxygen. Dumas (*Ann. d. Chim. Phys.*, 1878, xiv., 289) found 0.008 per cent of oxygen in similar silver by fusion *in vacuo*, and Mallet (*Phil. Trans.*, 1880, clxxi., 1020) found that silver fused like Stas's purest in the oxyhydrogen blast contained 0.005 per cent of oxygen).

Again, it showed that at least a part of the difference between the two new values for the atomic weight of sodium is due to an error in the assumed atomic weight of chlorine used in the calculations. It proved that the analytical method was capable of yielding very constant results with a given sample of silver (Exps. 72–76). It confirmed previous results in showing that portions fused in these various ways were not very different, but showed slight variations according to the method of fusion employed (see Richards, *Proc. Am. Acad.*, 1893, xxix., 65). It furnished a means of determining, by later comparison of one or the other of these results with those from the purest silver, the exact grade of purity of the various specimens of silver used in the previous work in this laboratory, in particular that used in the earlier part of the present research. Finally, it furnished sufficient clue to the precautions which must be taken to secure yet purer silver.

A careful study of the accompanying table led to the conclusion that a reducing environment is necessary to eliminate traces of oxygen during fusion, and that lime is better than carbon as a support during fusion. The details of this matter have already been described (p. 194) under the heading "Preparation of Pure Materials," and need not be further detailed here. Suffice it to say that the silver used in the experiments recorded in the table (Final Series) was free from the sources of error thus detected.

The Final Series comprehends ten experiments, made with four samples of silver prepared entirely independently by three different experimenters (Dr. Baxter having very kindly given us a piece of his purest silver used in the determination of the atomic weight of iodine, and each of the authors having made separate samples), and two entirely different methods of synthesis.

Of these methods of synthesis one has already been described, namely, the usual method of solution of the silver in nitric acid, precipitation by hydrochloric acid, and filtration on a Gooch perforated crucible. Of course all the precautions and corrections already discussed were applied with as much care as possible. All the vessels used were afterwards washed with a little ammonia, and the washings tested nephelometrically in order to be sure that no silver was lost by adsorption on the vessels. By this method Experiments 69, 77, 78, 80, 81, R. 2, and R. 3 were made. Further unvaried repetition of this process seemed to promise no further light on the question.

Although this method seemed to leave little or nothing to be desired as regards accuracy, it was nevertheless deemed advisable to test it by making a few syntheses by means of entirely a different process, in which all the vessels coming in contact with the precipitate could be weighed. Accordingly, a new method was devised, a modification of one of those used by Stas, employing quartz vessels instead of glass. A quartz dish was accurately weighed by substitution of a counterpoise of a similar composition, surface area, and weight. A watch-glass to serve as its cover was weighed in a similar fashion separately. A weighed piece of silver was placed in the dish, which was supported in a large empty "desiccator," whose bottom and sides were well moistened with pure water. The silver was covered with re-distilled nitric acid of the usual strength, and left covered both by watch-glass and desiccator lid to dissolve slowly over-night in a warm place. On the next day the moisture on the glass cover was washed, with great care to avoid the slightest spattering, into the quartz dish. If any spattering occurred it was caught in the large desiccator, which, having been tightly covered, served also to catch all spray proceeding from the solution of the silver. In two experiments, the silver recovered from the water in the desiccator amounted to only 0.02 m.grm. when estimated by the nephelometer, and in one no silver at all was found in the desiccator.

Hydrochloric acid gas was next allowed to play upon the surface of the silver nitrate solution in the quartz dish, and the resulting chloride was caused to sink to the bottom by means of a very small glass stirrer, afterwards carefully rinsed. The remaining solution of nitric and hydrochloric acids was then evaporated to dryness upon a clean steam-bath under an appropriate hooded cover (Victor Meyer funnel). The atmosphere around was kept as pure as possible during this evaporation, which was usually conducted during the night. Finally, the silver chloride was fused in the quartz dish supported inside of a large porcelain crucible from which the products of combustion of the gas-flame were suitably deflected. The weighed cover caught the one or two minute drops projected upward during the act of fusion. Then a mixture of chlorine and hydrochloric acid gas was passed in, so as to convert any residual traces of nitrate of silver wholly into argentic chloride. The precaution of cooling mentioned above enabled us to preserve the quartz dish intact through the analyses, notwithstanding the extreme fragility of thin fused quartz. Its weight was conserved through three experiments within the limits of the accuracy of the weighing. Experiments 83, 84, and 85 were made by this method. One of these is given below in detail as an example of the method.

Experiment 84.

	Grms.
Corrected weight of silver in air	6.25336
Vacuum correction	0.00018
Weight of silver in vacuum	6.25318
Excess weight quartz dish over counterpoise ..	0.21510
Excess weight dish and fused AgCl over counterpoise	8.52279
Weight of argentic chloride in air	8.30769

Excess weight cover glass over counterpoise ..	0.00972
Excess weight cover and spattering over counterpoise	0.00974
Spattering	0.00002
Weight argentic chloride in air	8.30771
Vacuum correction	0.00061
Weight of argentic chloride in vacuum ..	8.30832
AgCl found in desiccator by nephelometer ..	0.00002
Total weight of argentic chloride	8.30834
AgCl = $6.25318 : 8.30834 = 100.000 : 132.866$.	

This second method, which avoids any transference of material whatever, is an unusually searching check upon the method employing the Gooch filter, and that the two methods confirm each other to the limit of weighing is a most satisfactory criterion of each, supporting the previous similar work with sodic chloride as well.

The Final Series is complete, including all the experiments made with the purest material, and every precaution. The experimental work, as well as the preparation work, was the product of more than one experimenter, the senior author having made, from beginning to end, the syntheses numbered R. 2 and R. 3 (as well as R. 1 in the Preliminary Series), and the junior author having made all the others. The two experimenters used different balances, different weights separately standardised, and different preparations throughout, in order to secure additional certainty.

(The hydrochloric acid used in Experiments R. 1, R. 2, and R. 3 was three times successively treated with small amounts of permanganate, and boiled to eliminate bromide, and each time distilled).

For No. 69, Baxter's silver fused on lime in hydrogen was used; for Nos. 77, 78, 80, 81, 83, R. 2, sample T, fused in the same way; for R. 3, sample V, fused on lime in a vacuum, and for 84 and 85 sample W, fused on lime first in hydrogen and then in a vacuum.

The result, namely, 132.867 parts of the chloride from 100,000 of silver, has a "probable error," computed according to the method of least squares of about 0.0005. This so-called probable error of course gives no clue as to possible constant errors involved. A better idea of the chemical trustworthiness of the result is got by comparing the data furnished by the two different methods of synthesis. The averages of the seven determinations by the method of filtration is 132.8666, while that of the three by the method which involves no transference of material (83, 84, 85) is 132.8673. These are both within the range from the mean 132.8668 indicated by the "probable errors" of the respective averages, and hence may be considered as identical.

If the comparison be made according to the environment of the silver during fusion, a similar result is obtained. The silver fused in hydrogen gives an average of 132.8668, while that fused in a vacuum gives 132.8668. Again this is essential identity.

In view of these facts, the fact that all probable errors tend to lower the result, and the multitude of precautions which eliminated error from these figures, it is not unsafe to conclude that 100,000 parts of the purest silver really yields as much as 132.867 parts of argentic chloride. This conclusion confirms the comparison of the silver and argentic chloride indicated by the work on sodium, and shows that no great occlusion of sodic nitrate could have taken place there. It furnishes, moreover, a means of determining by comparison the purity of any other specimen which has been used in the quantitative synthesis of argentic chloride by either of the foregoing methods. For example, it shows that the silver used in the work on sodium—silver which yielded 132.862 parts of chloride—must have contained $132.862 = 132.8668 - 0.00037$ per cent of impurity.

(See Syntheses 65, 66, and 70 given in the table. The silver used in these syntheses was exactly similar to some of that used in the work on sodium. In the same way, if Stas's method of synthesis is considered as comparable with ours, his silver must have contained $(132867 - 132848) \div 132867 = 0.015$ per cent of impurity. It is doubtful, however, if the methods of synthesis are strictly comparable).

(To be continued).

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances de l'Académie des Sciences. Vol. cxlii., No. 20, May 14, 1906.

Conductivity of Ammonium Sulphate in Mixtures of Sulphuric Acid and Water.—G. Boizard.—When ammonium sulphate is dissolved in mixtures of varying proportions of sulphuric acid, and water, solutions are obtained whose conductivity is greater or less than that of the solvent. As a result of his experiments on this subject, the author asserts that the phenomenon shown by ammonium sulphate is general. This he proves by dissolving in mixtures of sulphuric acid and water—(1) All the sulphates, whether anhydrous or hydrated; (2) the bisulphates; (3) the mineral acids and a number of organic acids; (4) such salts as MnO_4K , $\text{PO}_4\text{H}(\text{NH}_4)_2$, NaCl , NO_3K , $\text{CH}_3\text{CO}_2\text{Na}$, &c.

Total Synthesis of Camphor Derivatives. Iso-lauroleone and Isolaurolic Acid.—G. Blanc.—The author completes the synthesis of the camphor derivatives, and states that this has only become practicable since the execution of the total synthesis of the $\alpha\alpha$ -derivatives of dimethyladipic acid, and also by certain improvements in the methods which he devises.

α -Chlorocyclohexanone and its Derivatives.—L. Bouveault and F. Chereau.—When chlorine acts on cyclohexanone, an α -chloro-derivative is obtained, but the hydrochloric acid which is formed at the same time causes the production of certain condensation products. The best results are obtained by performing the chloruration in presence of calcium carbonate in water. The results are still better if cyclohexanol is used instead of cyclohexanone, but a double quantity of chlorine must be employed. The α -substituted homologues of cyclohexanone can thus be prepared without difficulty, e.g., α -methylcyclohexanone, which boils at 160° under 10 m.m. pressure, its semicarbazone melting at 195° . α -Ethylcyclohexanone boils at 65° under 10 m.m. pressure, and its semicarbazone melts at 157° ; also α -isopropylcyclohexanone, boiling at 80° under 10 m.m. pressure.

Stereoisomerism in the Group of the $\alpha\beta$ -Acyclic Non-saturated Acids.—E. E. Blaise and P. Bagard.—The investigation and isolation of the non-saturated stereoisomeric acids has hitherto presented great difficulties—the majority of chemical, and even physical, agents transforming the labile isomers into the stable isomers. The authors now succeed in overcoming this difficulty and utilising M. Bodroux's reaction. They find that the ethers of the labile acids give the corresponding amides without transposition when they are treated with the bromo-magnesium derivatives of the amines. These amides, which easily crystallise and can be separated, can be used for a precise investigation and a certain identification.

Berichte der Deutschen Chemischen Gesellschaft.
Vol. xxxix., No. 7, 1906.

Two Acid Sodium Sulphates.—J. D'Ans.—Tri-sodium hydrosulphate, $\text{Na}_3\text{H}(\text{SO}_4)_2$, may be prepared by evaporating a solution containing equimolecular quantities

of sodium sulphate and sulphuric acid, when crystalline needles separate out on cooling. When a solution containing 16.5 per cent of sulphuric acid and 35 per cent of sodium sulphate is warmed, and sodium sulphate and sulphuric acid are added gradually, in the proportion of 10 grms. of the salt to about 2 c.c. of concentrated acid, crystals of $\text{Na}_3\text{H}(\text{SO}_4)_2 \cdot \text{H}_2\text{O}$ separate out. Their formation is accelerated by the addition of a crystal to the solution.

Dissociation of Barium Carbonate.—Alexis Finkelstein.—The study of the dissociation of barium carbonate has been found very difficult, because high temperatures have to be employed, and baryta when hot attacks the material of which the vessels containing it are made, such as platinum, porcelain, and all silicates; the author, however, has found that the exclusion of oxygen stops the action, and that in pure nitrogen baryta and platinum do not act on one another. From the curves he obtained it appears that the dissociation of barium carbonate occurs in two phases; first a basic carbonate is formed; this is easily fusible, melting below 950° . Its composition appears to be $\text{BaCO}_3 \cdot \text{BaO}$, and it is capable of dissolving BaO and also BaCO_3 at a higher temperature. This compound then decomposes into BaO and CO_2 . Barium carbonate melts at a temperature above 1350° . The heat of the dissociation reaction does not vary with the temperature.

Elementary Analysis with the Dennstedt and Heraeus Furnaces.—D. Holde.—The author has investigated the question of the advantages to be gained by the use of electric instead of gas furnaces for elementary analysis. Using a Heraeus furnace he finds the cost considerably greater than with a glass furnace, but the electric apparatus requires no special combustion room as the surroundings are not heated. The operation is as trustworthy as with gas and takes the same time, but need not be watched so long, while one great advantage is that the temperature of the air round the furnace is not raised, so that the experimenter suffers no inconvenience. With the Dennstedt furnace the combustion can usually be accurately performed in from fifteen to twenty-five minutes, except in the case of certain substances, but the disadvantage of the furnace is that during the quick analyses it must be watched incessantly.

Action of Light on Uranyl Acetate.—A. Bach.—When carbon dioxide is led through a solution of uranyl acetate exposed to direct sunlight the salt is reduced to a mixture of uranous and uranic hydrates. The salt suffers no such change if it is exposed to light in the absence of carbon dioxide, or if carbon dioxide is introduced in the dark. This result has been disputed by Euler, but the author has repeated the experiments and confirmed his conclusions. He finds that if the illumination, concentration of uranyl acetate, and diameter of the tube in which the salt solution is placed are kept constant, the time taken for the appearance of a definite turbidity is inversely proportional to the height of the liquid, and when the ratio between this height and the diameter has reached a certain value the reduction ceases. When illumination, diameter, and height of column of liquid are constant, the time of reaction varies inversely as the concentration of the solution. Hot saturated solutions are almost instantaneously reduced.

New Method of Preparing Ketones.—Hugo Haehn.—Commercial calcium carbide reacts in the cold with any fatty monocarboxylic acids, yielding acetylene. When this reaction takes place at a higher temperature the ketones may be directly obtained, two molecules of acid giving off water and carbon dioxide, $\text{R} \cdot \text{COOH} = \text{R} \cdot \text{CO} \cdot \text{R} + \text{CO}_2 + \text{H}_2\text{O}$. The water acts on the carbide, yielding acetylene and calcium oxide, or the hydrate is left behind. A combustion tube is filled with pieces of calcium carbide as large as a pea; the acid is allowed to drop slowly into one end of the tube, which is heated in a combustion furnace, and the ketone is collected

in a receiver at the other. The raw ketone should be neutral. If it has an acid reaction either the distillation is proceeding too quickly or the temperature of the carbide is not high enough.

MISCELLANEOUS.

The Electrician.—Mr. W. R. Cooper, M.A., B.Sc., M.I.E.E., A.M.Inst.C.E., has been offered and has accepted the position of Editor of *The Electrician* in the place of Mr. F. C. Raphael, A.M.I.E.E., who retires on June 30th.

Literary Note.—Messrs. Archibald Constable and Co. will shortly publish "Chemistry of Paints and Paint Vehicles," by C. H. Hall. The book is written from the standpoint of a Chemist actively employed in the manufacture of Paints and Colours, and it is a practical book for practical men.

MEETINGS FOR THE WEEK.

WEDNESDAY, 20th.—Microscopical, 8. "On the Structure of some Carboniferous Ferns," by the President.

THURSDAY, 21st.—Chemical, 8.30. "Cleve Memorial Lecture," by Prof. T. E. Thorpe. "Constituents of the Essential Oil from the Fruit of *Pittosporum Undulatum*," by F. B. Power and F. Tutin. "Mobility of Substituents in Derivatives of β -Naphthol," by J. T. Hewitt and H. V. Mitchell.

FRIDAY, 22nd.—Physical, 5. "Effect of Radium in Facilitating the Visible Electric Discharge in *vacuo*," by A. A. Campbell Swinton. "Comparison between the Peltier Effect and other Reversible Heat Effects," by A. O. Allen. "Effect of the Electric Spark on the Activity of Metals," by T. A. Vaughton. "Dielectric Strength of Thin Liquid Films," by P. E. Shaw. "Effect of Electrical Oscillations on Iron in a Magnetic Field," by W. H. Eccles.

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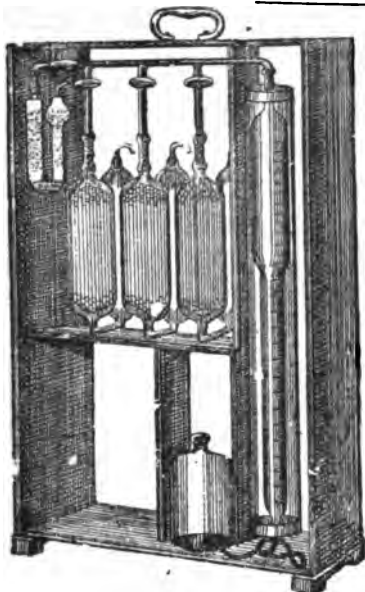
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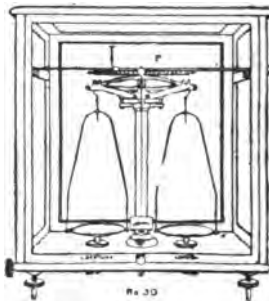
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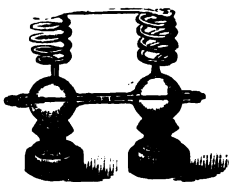
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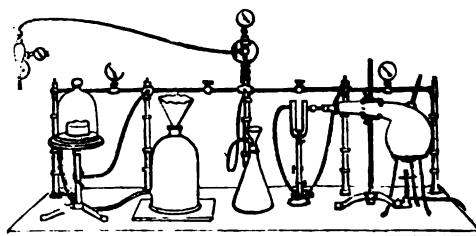
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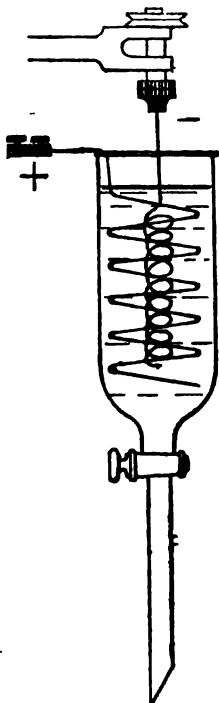
A SIMPLE FORM OF ROTATING ELECTRODE FOR ELECTRO-CHEMICAL ANALYSIS.*

By F. MOLLWO PERKIN, Ph.D.

At the first meeting of the Faraday Society I published a note upon a rotating electrode for electro-chemical analysis (*Electrochemist*, iii., 26). It consisted of a sand-blasted gauze cylinder fastened on to the end of a stout iridio-platinum wire, and which was rotated within a circular anode. The anode was made of two rings of thick platinum wire which were connected together by four small baffles. The object of the baffles was to prevent the liquid being swirled round in the electrolysing vessel by the rapid rotation of the cathode. The results obtained with this electrode were quite satisfactory, and in their paper on the "Electrolytic Deposition of Zinc," Dr. Price and Mr. Judge have also obtained very satisfactory results with this form of cathode.

As it is always an advantage to use the simplest possible apparatus for analytical purposes, more especially apparatus which can readily be obtained or at any rate constructed in any laboratory, it occurred to me to endeavour to simplify the cathode for rotation work. The cathode as originally described could not be made in the laboratory, but the one which I have recently adopted can be made without any difficulty.

As will be seen from the diagram the cathode simply consists of a spiral of platinum wire. It is, however, best to use iridio-platinum wire because this is much more rigid and the electrode is less likely to "throw out" when rotated at high speeds. The electrode originally employed was made of ordinary platinum wire, 1 m.m. in diameter. The ones at present in use are made from an alloy of platinum with 20 per cent of iridium. It must be admitted that as this alloy is very hard it is not easy to coil into a spiral, and it is therefore better to get the makers (Messrs. Johnson, Matthey, and Co.) to make the spiral when ordering the wire. It is a further advantage to have the spiral sand-blasted; for a great many metals, however, this is unnecessary. The total active surface of the electrodes now in use is about 23 sq. c.m. The anode consists of a 1 m.m. platinum wire wound into two or three spirals so as to fit tightly against the electrolysing vessel. It will be noticed that I use the same apparatus, viz., a tap funnel, for holding the solution to be analysed as that described by Dr. Price and Mr. Judge, and I am indebted to them for the suggestion. This form of electrolysing cell is extremely useful and very simple, and is of great advantage for washing the deposited metal without stopping the current or removing it from position.



Below is appended the results obtained with this form of cathode in the analysis of copper.

CuSO ₄ taken.	Current in amperes.	E.M.F.	Time in minutes.	Deposited copper.
0.0506	0.45—4.8	2.7—6.0	35	0.0498
0.1013	0.6—2.0	2.8—4.8	38	0.1005
0.1266	1.0—5.5	3.0—9.0	32	0.1253
0.1013(a)	0.5—2.0	5.0—7.6	40	0.1009
0.1266	0.45—3.0	—	40	0.1266
0.1266	0.5—4.5	—	35	0.1263

(a) From cyanide solution.

The bulk of solution was in all cases about 50 c.c., and the quantity of concentrated nitric acid added from 0.5 to 1 c.c.

Owing to the extremely high cost of platinum, one would often like to employ less expensive metals for cathodes. I might say that a spiral of nickel wire can quite satisfactorily be employed for a good many metals; for example, for copper, zinc, or iron. The metals are readily removed by means of, in the case of copper, moderately strong nitric acid, and it is surprising how little the nickel itself is acted upon by this acid. In the case of zinc or iron dilute hydrochloric or sulphuric acid may be used, and also against these acids the nickel is very fairly resistant. Of course, when nickel electrodes are used it is necessary to weigh them after each determination before they are again used. With platinum this is not absolutely essential, but it is always a wise precaution to take. I have an electrode of nickel wire which has been used fifteen times for copper and nickel determinations, and it looks as if it might be used for quite another forty such determinations.

Platinum is usually regarded as a metal which is practically unacted upon by reagents, with the exception of aqua regia. When employed in electro-chemical work as an anode, however, it is by no means so stable as it is generally considered; this is especially the case when high current densities are employed. For example, in potassium cyanide solution the platinum is distinctly soluble, as the following numbers will show. A weighed anode of platinum wire having a surface of about 10 c.m. was placed in a 4 per cent solution of potassium cyanide, and a current of 4 amperes passed for thirty-five minutes. At the end of the time the platinum had lost 0.0016 gm. It was again placed in a the solution, and a current of 3 amperes passed for fifty minutes; the loss in weight was now 0.0015 gm. In this connection it should be of interest to electro-chemists to hear that Messrs. Johnson, Matthey, and Co. are now supplying iridium of over 99 per cent purity. I am informed that this is the first occasion that the metal of such a high state of purity has been obtainable. The metal is as hard as steel, and may be boiled in aqua regia without being attacked; it is also unacted upon by molten lead. I understand that the fact of its not alloying with lead is made use of in its preparation. It has not been found possible to draw it into wire, and it can only be rolled into sheets when hot.

Direct Oxidation of Cæsium, and certain Properties of Cæsium Peroxide.—E. Rengade.—It is known that cæsium burns immediately on contact with air. The author investigates the question as to whether this property is due to the presence of water, and also how the metal behaves with regard to pure dry oxygen. As a result of his experiments, he finds that even perfectly dry oxygen does attack cæsium at ordinary temperatures with great energy, and more slowly as the temperature is reduced. The action of excess of oxygen on cæsium causes the formation of the yellow peroxide, Cs_2O_4 , which is easily dissociable, and is decomposed by water at ordinary temperatures, and reduced by carbon dioxide and by hydrogen at temperatures very little above normal.—*Comptes Rendus*, cxlii., No. 21.

* A Paper read before the Faraday Society, June 12, 1906.

RELATIVE STRENGTHS OF ACIDS.

By PHILIP BLACKMAN.

ARRHENIUS showed that if acids be arranged in the order of their relative strengths they were also arranged in the order of their molecular conductivities (W. Ostwald, "Lehrbuch der Allgemeinen Chemie," II., i., 650). The comparison in practically all cases is so remarkably close that the electrical conductivity method has been accepted as a means for determining the relative strengths of acids. A satisfactory explanation (on the hypothesis of the "degrees of dissociation of acids") has been advanced to establish the connection between the relative strengths of acids and their molecular conductivities, and in this paper an attempt is made to supply a proof.

It was shown by the author ("Quantitative Relation between Molecular Conductivities," *Phil. Mag.*, 1906, xi., 416) that $\mu_{vHX} + \mu_{vM_1OH} - \mu_{vM_1X} = K$ (a constant); where μ_{vHX} , μ_{vM_1OH} , and μ_{vM_1X} represent respectively the molecular conductivities (measured at the same molecular concentration, v , and at the same temperature) of the acid HX , the base M_1OH , and the salt M_1X . This value K represents the loss of electrical conductivity corresponding to the equation $H + X' + M_1 + OH' = H.OH + M_1 + X' + K$ or $H + OH' = H.OH + K$. It is reasonable to conclude that, had there been in the final solution H and OH' ions (i.e., $H + OH' = \text{not } H.OH$, but $H + OH'$), the value of K would have been equal to zero. In other words, K would represent the molecular conductivity of water—under the conditions mentioned, viz., (1) temperature and (2) molecular concentration.

It was shown that K is a constant for each series only, but is not a common constant for all series, i.e., K varies with the nature of the acid. This can be explained in one of two ways, viz., either (1) the "molecular conductivity" of water is a variable quantity, or (2) the "molecular conductivity" of water is really a constant, the variability being due to the fact that the stronger the acid is the greater does the value of K become, the maximum relative value (at any one temperature and concentration) being reached in the case of the strongest acid. Of these two assumptions, the first is improbable and the second is apparently correct.

Assuming, then, that the molecular conductivity of water is constant, and also assuming for the moment that all acids were of equal strength, it would be a necessary condition that—

$$\begin{aligned}\mu_{vHX} + \mu_{vM_1OH} - \mu_{vM_1X} &= K_{(HX)} \\ &= \mu_{vHX_1} + \mu_{vM_1OH} - \mu_{vM_1X_1} = K_{(HX_1)} \\ &= \mu_{vHX_2} + \mu_{vM_1OH} - \mu_{vM_1X_2} = K_{(HX_2)} \\ &= \dots\dots\dots = \dots\dots\dots \\ &= \text{constant}\end{aligned}$$

(supposing that the terms be arranged so that $\mu_{vHX} > \mu_{vHX_1} > \mu_{vHX_2} > \dots\dots$). In practice, however, this constancy is not observed, as acids are not all of equal strength. The equations may, nevertheless be rendered mathematically equal in several ways, the most useful of which is that which will yield quantitative comparative results. By introducing the factors $x_1, x_2, \dots$ and $y_1, y_2, \dots$ respectively, such that $\mu_{vHX} = x_1 \cdot \mu_{vHX_1} = x_2 \cdot \mu_{vHX_2} = \dots$ and $\mu_{vM_1X} = y_1 \cdot \mu_{vM_1X_1} = y_2 \cdot \mu_{vM_1X_2} = \dots$, the above equations become mathematically true. Bearing in mind that the greater the equalising quantities $x_1, x_2, \dots$ are, the smaller must be the respective strengths of the acids, it is at once evident that the relative strengths of the acids $HX, HX_1, HX_2, \dots$ are respectively—

$$\frac{1}{x_1}, \frac{1}{x_2}, \frac{1}{x_3}, \dots;$$

or, expressed as percentages,—

$$100, 100/x_1, 100/x_2, \dots$$

But—

$$x_1 = \mu_{vHX} / \mu_{vHX_1}, \quad x_2 = \mu_{vHX} / \mu_{vHX_2}, \dots;$$

consequently, the relative strengths of the acids $HX, HX_1, HX_2, \dots$, expressed as percentages, are respectively—

$$100, 100 \cdot \mu_{vHX_1} / \mu_{vHX}, \quad 100 \mu_{vHX_2} / \mu_{vHX}, \dots$$

East London Technical College, London, E.

INVESTIGATIONS ON THE OCCURRENCE OF CHANGES IN THE TOTAL WEIGHT OF SUBSTANCES UNDERGOING CHEMICAL TRANSPOSITIONS.*

(SECOND COMMUNICATION).

By H. LANDOLT.

(Continued from p. 274).

PROCESS.

THE experiments were performed in practically the same way as the earlier ones. The following information may be given concerning the details of the methods used:—

1. Reaction Vessels.

For most of the experiments η -shaped tubes of Jena apparatus glass were used; their limbs were 10 c.m. long and 5 c.m. across. On the upper bent connecting piece, of length 8 c.m. and diameter 2 c.m., were fixed the two short filling tubes 0.7 c.m. wide. Weight of the full apparatus, 360 to 450 grms.; external volume, 400 to 450 c.c.; external glass surface, 370 to 400 sq. c.m.; substances introduced, inclusive of water, 250 to 350 grms. The η -tubes were smaller than those used in the old experiments, when their weight was 700 to 980 grms. and their volume about 900 c.c.

A second kind of apparatus, denoted hereafter by O, consisted of a glass cylinder (A), 12 c.m. high and 7 c.m. wide, closed at the bottom and ending in a filling tube at the top. In the interior of A, at the bottom, an uncovered glass beaker (B) was fused; its height was 8 c.m. and width 5 c.m. Thus a circular space was formed, in which was placed one of the reacting substances, while the other was put inside the beaker B. Finally, the cylinder was surrounded by another larger closed Dewar's glass enveloping vessel C, 13 c.m. high and 8 c.m. across, the space between A and C being exhausted. Thus the volume of the outer vessel C is unaltered by the changes of volume which the vessel A might undergo in consequence of the heat of the reaction. Weight of apparatus when filled, 450 to 550 grms.; contents, 170 to 260 grms.; external volume, about 600 c.c.; external surface of the glass, about 350 sq. c.m.

Thirdly, I used for some experiments η -shaped vessels of quartz glass, which had been made by Herr Heraeus, of Hanau. They were the same size as the η -tubes of glass, but possessed only one opening for filling, at the highest point of the arch. This was originally closed with a fused mixture of three parts of colophony and one part of wax, and was afterwards closed by fusing in the oxy-hydrogen blowpipe. The vessels have not as yet been much used, because, on account of the great thinness of their walls, there was a fear that an alteration of pressure in the interior would have an effect upon their volume.

Finally, η -tubes of glass were also used, the inner walls of which were covered with a layer of solid paraffin. I was induced to use these because both in the older and more recent experiments I had observed that glass vessels sometimes do not appear to be perfectly impermeable, either in consequence of a little flaw or of an air-bubble in the glass. The presence of such places is noticed in taking the first series of weighings, if the difference of weight of the two vessels undergoes a slight change of the

* From *Sitzungsber. d. K. Akademie d. Wissensch.*, 1906, viii., 266.

same sign from day to day.* The same penetrability, which causes much waste of time, has also been observed by Heydweiller.

All the glass vessels were allowed to lie for several days in dilute sulphuric acid, and then in ammonia, to make their external layer less rich in alkali, and therefore less hygroscopic.

2. Preparation and Adjustment of the Vessels.

When filling the Π -tubes with the substances, care was taken to load each limb equally. If this was not possible, as, for example, in experiments on the solution of salts in water, the equalisation was effected by the addition of indifferent substances, such as Bohemian garnets. In most cases, moreover, the liquid surface was covered in one or both limbs of the Π -tubes with a layer of paraffin-oil, in order to prevent evaporation.

After fusing up both the reaction vessels, their weight was determined by means of a kilogram balance which was sensitive to 1 m.grm., and their volume was ascertained by weighing in water at the same temperature. Then for the smaller and lighter vessel a closed addition-body was made of thin glass tubing of diameter 5 m.m., and its volume was altered by immersion in a measuring tube partially filled with water, by means of which 0.02 c.c. could be read off, until the two vessels were adjusted to less than 0.04 c.c. The error caused by the deviations of the weight of the air between the limits 1.15 and 1.25 m.grms. per c.c. thus lay below 0.004 m.grm. The adjustment of the weight to a few m.grms. was performed partly by adding sand to the addition-body and partly by hanging on platinum-wire.

The following is an example of the adjusted apparatus:—

	Vessel A.		Vessel B.	
	Wt. (grm.).	Vol. (c.c.).	Wt. (grm.).	Vol. (c.c.).
Original vessel .	352.285	393.133	345.019	390.459
Addition body..	—	—	7.566	2.678
Platinum wire .	0.159	0.007	0.155	0.007
	352.744	393.140	352.740	393.153

Difference of weight, 0.004 m.grm.; difference of volume, 0.013 c.c.

For the weighing, the Π and O vessels were stored in equally heavy stands of gilded brass, which raised the load on the balance pans to about 490 to 550 grms. In moving the vessels touching with the hand was avoided, an instrument like a fork of polished steel being used. For the purpose of carrying out the reaction, the apparatus was placed on a suitably constructed brass stand, which touched the first only in a few places, and the mixture of the two substances was effected by gradually inclining the whole arrangement.

3. Balance.

The instrument used was made in the workshop of Alb. Rueprecht, of Vienna, who is famous for the construction of precision balances.† It was capable of carrying 600 grms.; length of brass beam 150 m.m.; end planes connected by means of two crossed knife-edges with the scale suspension; platinum weights of 120, 121, 122, 140, 155.5, 160 m.grms., hung on from outside; mirror and telescope reading to a distance of 3 metres; moreover, the loads could be automatically altered and transported on to the pans by poles of equal length, and, finally, the beam and pans released; sensitiveness for 1 m.grm. at a load of 500 grms., originally 400 scale divisions (tenths of a m.m.), after four years' use, gradually reducing to 280; half the time of oscillation, thirty-five seconds. The balance was placed in a room of the former II. Chemical Institute of the University, and was there exposed but little to disturbances from the street, though a good deal to those from the interior of the building, which, however, could be prevented. The room was heated by a gas-

furnace always burning, with an ethyl chloride regulator, which enabled a perfectly constant temperature to be maintained.

The determination of the difference of weight of the two vessels was performed by Gauss' method, given on p. 310 of the first paper, the loads being exchanged twice and the sensitiveness determined four times. Thus, altogether four oscillation points had to be determined, each of them by releasing the beam three to five times. The details of the method will be explained in the complete article. I will here confine myself to some details of the performances of the balance.

(a) When two cylindrical 400 grms. brass weights, differing from one another by about 4 m.grms., were placed in exactly the same position on the pans, and weighed on different days, the exact differences of weight were:—

4.2588, 4.2591, 4.2589, 4.2584, 4.2576 m.grms.; mean, 4.2586; mean error, ± 0.0003 m.grm. (The mean, and not the probable error, is calculated in the following determinations).

Greatest difference in weighing, 0.0015 m.grm.

(b) In another series of weighings of the same weights the deviations were, however, much, greater, e.g.:—

4.273, 4.260, 4.250, 4.242, 4.260 m.grms.; mean, 4.257 ± 0.005 m.grm.

Greatest difference in weighing, 0.031 m.grm.

(c) The determinations of the differences of weight of the glass reaction apparatus gave the following results:—

I. 3.964, 3.972, 3.958, 3.971, 3.966 m.grms.; mean, 3.966 ± 0.004 m.grm.

Greatest difference in weighing, 0.014 m.grm.

II. 5.816, 5.804, 5.793, 5.839, 5.827 m.grms.; mean, 5.816 ± 0.008 m.grm.

Greatest difference in weighing, 0.046 m.grm.

4. Errors in Weighing.

These were chiefly the results of the following influences:—

(a) *Different Temperature of the Two Beam Arms.*—To ascertain this more exactly, through the copper covering plate of the balance two thermometers were introduced, the large mercury vessels of which were directed towards the middle of each half of the beam and were some m.m. away from it. The projecting scales were read by means of a telescope, being temporarily illuminated from behind for this purpose by means of an incandescent lamp. The thermometers, with which $\frac{1}{100}^{\circ}$ could safely be estimated, agreed very well, and during a weighing no difference could ever be observed between them. Nevertheless, there were possibly differences of thousandths of a degree, and as calculation shows, if the length of the beam arm is 150 m.m., the load 500 grms., and the coefficient of expansion of brass 0.000019, a difference of temperature of 0.001 $^{\circ}$ would alter the result of the weighing by about 0.01 m.grm. To ensure uniform distribution of heat, in the interior of the balance on the back wall a thick copper plate was placed, and the case was surrounded by screens as well.

(b) *Uniform Alteration of Temperature of the Two Beam Arms.*—If the load was weighed on different days at varying temperatures it always appeared that increase of heat produced a considerable displacement of all the oscillation points towards the right, and this amounted to about 10 whole, equivalent to a reading of 100 scale divisions for 1 $^{\circ}$. The left half of the beam must thus expand more than the right. Many determinations of weight, performed at different temperatures, which, however, remained constant during the weighing, gave concordant results. As, however, the whole process of weighing took from one hour to an hour and a-half, sometimes it was unavoidable that the two thermometers uniformly rose or fell some hundredths of a degree. In this case, when Gauss' method was used, the heat effect was compensated, yet the small changes of temperature caused an incipient disturbance of the balance, in consequence of

* Examples of such steady changes are given in the first paper, p. 314.

† It is shown in the price list of this firm for 1902, p. 29, No. 48.

which on repeated releasings greater differences appeared in the equilibrium positions.

(c) *Dissimilar Positions of the Loads on the Scale Pans.*

—If the load is not distributed perfectly uniformly round the centre of gravity line from the end knife-edge of the beam, on releasing the balance a shifting of the scale pan with its suspension will take place; this causes an inclination of the planes towards the knife-edges, which are not absolutely sharp, and thus a slight change is produced in the length of the beam. If this amounts to only 0.0001 m.m., and the length of the beam is 150 m.m., and the load 500 grms., the effect upon the result of the weighing will be 0.333 m.grm. In Rueprecht's balance this error is for the most part obviated by the insertion of two crossed knife-edges between the plane suspension and the scale bow; nevertheless, it is necessary to place the load, *i.e.*, the reaction vessel with its stand, as centrally as possible. A special instrument was used for this purpose. It consisted of a balance pan capable of turning vertically, and having on its lower side a point 10 c.m. long, which played against a second point on the pedestal of the arrangement. After the apparatus had been placed by means of guiding pins always in the same central position on the scale pan either by moving the glass vessel on its stand, or, after the reaction had taken place, by pouring the liquid from one limb of the Π -tube into the other, the mass had to be distributed so that when the pan turned the two points always fell together. Then the two pieces of apparatus were brought into the balance, when the mechanism acted so that they were always placed in the same position on the balance pans. They could also be placed in the same position turned through 180°. If they had been arranged centrally the weighings in the two positions agreed very well. If the masses were not distributed symmetrically, on the other hand, differences of nearly 0.1 m.grm would appear, but in this case satisfactory results could be obtained if the two pieces of apparatus were placed in each of the two positions, and the mean of the four weighings taken. This will be treated more fully in the detailed article.

Other influences, such as electrical or magnetic, were not taken into consideration.

5. *Errors caused by the Reaction Vessels.*

These might be due to the following causes:—

(a) *Warming of the Vessel when the Reaction Occurred.*

—The mixing of the substances was never performed suddenly, but in small portions, lasting two days. However, sometimes a rise of temperature of 5° to 7° was unavoidable. This might give rise to, firstly, an increase in the volume of the vessel, which possibly would not return to its original volume on cooling, and, secondly, a decrease of the water-film on the outer glass surface. As the vessel would lose weight for both these reasons, this was perhaps the simplest explanation of the many decreases of weight which have been observed in chemical reactions, and hence the effect of the warming had to be examined more closely. For this purpose one of two pieces of apparatus which had been weighed many times was placed in an air-bath heated to 30° or 40° for a quarter to one hour, and then after two days again weighed. Many experiments performed during my earlier work and again now showed that, even with this much greater warming, practically no alteration of weight could be proved. The proofs are given in the detailed article. Heydweiller (Drude, *Ann. d. Phys.*, v., 402, 403) has obtained the same result.

As regards the so-called water film on the outer surface of the glass, consisting of glass solution, condensed oil vapours from the machined parts of the balance, and particles of dust, the weight of it upon the surface of the Π -vessels, which amounted to 370 sq. c.m., lay between 0.133 and 0.182 m.grm. With the same sized but much less hygroscopic quartz vessels, it amounted to only 0.037 to 0.082 m.grm. These numbers were obtained from the loss of weight which an apparatus which had remained for several weeks in the balance suffered if it was wiped with a cambric cloth moistened with alcohol.

As in the reaction experiments two vessels of exactly the same surface were always used, the weights of the water film could not differ, and the constancy of the weights of the same apparatus when weighed for weeks showed that the alteration of it resulted in the same way from differences in the amount of moisture in the air.

(b) *Alteration of Volume of the Vessels caused by Alterations of Pressure inside them.*—The chemical reactions performed are always accompanied by a change of volume of the total mass, which is a decrease if from liquids solids separate, and *vice versa*. Thus, when a reaction occurs between silver sulphate and iron sulphate in aqueous solutions of the concentrations used, a decrease of the volume of the masses of the liquids occurs, amounting to 1.67 per cent. (Calculated from the densities of the solid substances and solutions before and after the changes). If the substance introduced into one apparatus besides the water has a volume of 300 c.c., this is reduced to 295 c.c. during the reaction, and if 100 c.c. of air at 760 m.m. pressure were above the liquid, the pressure would sink to 724 m.m. In order to find what effect such alterations of pressure exert on the walls of the Π -tubes, which were 0.75 to 0.8 m.m. thick, a special apparatus of the same glass was prepared. This consisted essentially of a closed cylindrical vessel of cubic contents 400 c.c., surrounded by a glass envelope filled with water which led to a narrow graduated tube. When the air in the inner cylinder is condensed or rarefied, the alteration of volume can be very plainly seen by the movements of the water in the tube, and many different methods showed that for each 100 m.m. increase or decrease of the pressure the original volume of 400 c.c. increased or decreased by 0.0036 c.m. As in the reaction experiments the variation of pressure never amounted to 100 m.m., the volume in the vessels altered so little that no error was to be feared in the weighing.

Some other possible causes of error which were investigated may now be discussed shortly. Thus, the fact that spots and sometimes flakes containing potassium cyanide appear on galvanically gilded brass surfaces owing to oxidation, induced me to examine for alteration in weight the gilded stands used to support the Π -vessels. It was found that the weight remained perfectly constant. As, moreover, as I have already mentioned some vessels filled with the reaction liquids showed decreases of weight on continued weighing without any apparent reason, I investigated whether possibly the particles of water slowly penetrated the thin glass wall. For this purpose one of the two vessels was filled with water and the other with paraffin-oil, and the differences in the weights determined during a period of three months. It was found that the vessel containing water became no lighter.

Almost all the sources of error enumerated have more or less influence upon the final result of the weighing, but they cannot be taken into account separately. In order to get some idea of their total effect a special series of observations was necessary, consisting in filling two vessels with quite indifferent substances, and subjecting them to the same operations and weighings as those filled with the reacting substances. This would give the error affecting the whole method. The first group of the following results deals with the determination of this error.

(To be continued).

Researches on Azoics. Transformation of the Orthocarboxyl Azoics into Chloro-oxyindazylic Derivatives.—P. Freundler.—The author has recently proved that the azoics which possess an ortho-substituted alcohol or aldehyde function can be transformed with the greatest ease into the corresponding indazylic derivatives. An investigation of azoics with an ortho-substituted radicle affords a still more curious example of this type of reaction. The author's experiments show the existence of an indazylic radicle on the one hand, and the presence of the OH group on the other hand.—*Comptes Rendus*, cxlii., No. 21.

A REVISION OF THE ATOMIC WEIGHTS OF SODIUM AND CHLORINE.*

By THEODORE WILLIAM RICHARDS
and
ROGER CLARK WELLS.

(Concluded from p. 280).

THE NEW ATOMIC WEIGHTS OF SODIUM AND CHLORINE, AND THEIR EFFECT ON OTHER ATOMIC WEIGHTS.

FROM the preceding description of the syntheses of argentic chloride, it is clear that 100·000 grms. of the purest metal yields 132·867 grms. of chloride. If then the atomic weight of silver is taken as 107·920† (a convenient value to assume for preliminary calculation), the molecular weight of argentic chloride becomes by simple proportion 143·393, and by difference the atomic weight of chlorine becomes 35·470.

(In this connection it should be pointed out that Leduc called attention to the fact that if Stas's silver really contained as much oxygen as Dumas said it must, chlorine would become 35·47; *Comptes Rendus*, 1901).

This value is about 0·05 per cent greater than the value announced by Stas; and the change produces a serious effect on a number of atomic weights. It is, of course, impossible to be perfectly certain of the accuracy of the new value, because it may contain concealed within it some entirely unsuspected source of error, but at least it is free from the real mistakes in the earlier work. It was reasonably certain that at least ten more syntheses would be needed to affect the value one unit in the third decimal place, if the further results varied no more widely than those recorded above in the final series. Hence it seems unlikely that chlorine is above 35·471 or below 35·469 if silver is taken as 107·920.

What, now, is the effect of this new value upon the atomic weight of sodium? In the table given on p. 238 it is shown that 100·000 grms. of argentic chloride could be obtained from 40·780 grms. of sodic chloride. If now argentic chloride is taken as 143·390, sodic chloride becomes 58·474, and by subtracting the new value for chlorine, 23·004 is obtained as the new value for sodium.

This is not, however, the only value for sodium which may be calculated from our results. On p. 261 has been given a table showing the results of ten experiments on the comparison of sodic chloride with silver direct, and on p. 276 the results of two more. These determinations were made before we had discovered the best method of obtaining pure silver; but the trace of impurity is allowed for in those tables, and it is safe to conclude that 100·000 parts of the purest silver are equivalent to 54·185 parts of sodic chloride. If, then, silver is taken as 107·920 and chlorine as 35·470, sodium becomes 23·007.

Thus two entirely independent values, calculated from two entirely separate series of experiments, yield respectively the values 23·004 and 23·007 for the atomic weight of sodium. Of these two, the latter is somewhat the more trustworthy, because, as has been said, the occlusion of sodic nitrate by the precipitate does not affect it; hence the mean may be taken as 23·006. This value is probably not more in error than two units in the third decimal place, if silver is 107·920, although of course the same remark applies to it that was made concerning the possible error of the value for chlorine. If silver is taken as 107·930, sodium becomes 23·008, and chlorine 35·473. The new values are, then, as follows:—

	Ag = 107·920.	Ag = 107·930.
Atomic weight of sodium ..	23·006	23·008
Atomic weight of chlorine ..	35·470	35·473

The new value for sodium is nearly 0·2 per cent less than that found by Stas—a percentage error much greater even than that made by him in the case of iodine. It will be

noted that the likely error of the new result is only about a twentieth of the difference between the new result and that of Stas. Some will attach significance to the fact that both the new values bring the elements nearer to the demands of Prout's hypothesis.

These new values affect greatly the figures in the second decimal place of all other atomic weights depending directly or indirectly upon chlorine, sodium, or silver. The number of elements thus affected is so great and the relations so complicated as to render imperative a re-calculation of all atomic weights. Nitrogen, as computed from ammoniac chloride, will be especially affected, the new value approaching more nearly that required by Avogadro's rule than the old. Probably, however, it will be best to delay this systematic re-calculation until a few other new data shall have been obtained—in particular new analyses of potassic chloride, argentic chlorate, the bromides, sulphides, and sulphates, and similar important compounds. Some of these are already under way at Harvard, and others will be undertaken at once.

In conclusion, it is a pleasure to acknowledge our great indebtedness to the Carnegie Institution of Washington, without whose liberal support the present investigation could not have attained its present thoroughness or precision.

SUMMARY.

The investigation consisted of a very careful quantitative study of three ratios—namely, AgCl : NaCl, Ag : NaCl, and Ag : AgCl. The effort was made to test every operation involved in the execution of the experiments. In the course of the work, the following points were developed:—

1. Sodic chloride, obtained from many sources and purified in many ways, always gave the same equivalent weight.

2. Fusion in vacuum of salt already fused in air caused no change in this equivalent weight.

3. Argentic chloride precipitated from aqueous solutions always occludes traces of other substances present, and those traces can not always be eliminated. Very dilute solutions must hence be used in precipitation.

4. The conditions governing the occlusion and release of these impurities were minutely studied, and it was shown that Stas's method of dropping solid salt into a silver solution causes occlusion of salt. Many considerations important in all precise chemical work are discussed.

5. A careful study of the solubility of argentic chloride was made, and the precautions necessary in using the nephelometer in the estimation of traces of chloride and silver were ascertained.

6. Fused argentic chloride probably contains traces of dissolved air, but not enough to affect essentially its weight, since subsequent fusion in vacuum caused no appreciable loss of weight.

7. The most difficult question in the purification of silver was found to be the elimination of the inclosed mother-liquor without introducing other impurities. Fusion on pure lime, first in pure hydrogen and then in a vacuum, is the safest method. Stas's silver must have contained at least as much oxygen as Dumas claimed.

8. In ten experiments, 44·5274 grms. of sodic chloride yielded 109·1897 grms. of argentic chloride.

9. In twelve other experiments, entirely distinct from these, 49·5007 grms. of sodic chloride were found to be equivalent to 91·3543 grms. of the purest silver.

10. In ten other experiments, again entirely distinct from the preceding, 82·6689 grms. of the purest silver yielded 109·8395 grms. of argentic chloride. Two very different methods of synthesis were used in this series, and the silver came from various sources, these variations being without effect on the result.

11. If the atomic weight of silver is assumed to be 107·920, sodium is found from the above results to have an atomic weight of 23·006, and chlorine an atomic weight of 35·470.

12. Many other atomic weights are affected, in their second decimal places, by these changes. In particular,

* Published by the Carnegie Institution of Washington, April, 1905.

† F. W. Clarke has for a long time accepted this value, and it is now assumed because it is probably nearer the true value than 107·93.

certain slight anomalies previously noticed in Harvard work are explained by them, and the atomic weight of nitrogen computed from ammonia is brought nearer to the value required by Avogadro's rule. Other anomalies appear in other places, however, and it is clear that many new atomic-weight investigations must be instituted to explain them, with due attention to hitherto unheeded dangers, especially of occlusion.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, June 7th, 1906.

Prof. R. MELDOLA, F.R.S., President, in the Chair.

MESSRS. E. Feilmann and H. G. Smith were formally admitted Fellows of the Society.

Certificates were read for the first time in favour of Messrs. Robert Anderson, Chester Road West, Sunderland; Reginald William Malyon Gibbs, B.Sc., Hetty Villa, Cam Road, Chesterton, Cambridge; Frederick James Martin, Passagem de Marianna, Minas, Brazil; Herbert Arthur Mills, Waverley, Bloomfield Gardens, Bath; Leonard Edward Beard Pearce, 79, Gordon Road, Ealing, W.; Thomas Ebenezer Pye, West View, Summersdale, Chichester; Frederick John Salmon, c/o Princess Estate and Gold Mining Co., Ltd., P.O. Box 112, Roodepoort, South Africa.

Of the following papers those marked * were read:—

*106. *Ammonium Selenate and the question of Isodimorphism in the Alkali Series.* By ALFRED EDWIN HOWARD TUTTON.

Normal ammonium selenate crystallises differently from the seven rhombic normal sulphates and selenates of the alkalis already investigated, namely, in monoclinic prisms or tables, which are described in detail in the paper. A rhombic form of the salt was once obtained (in 1852) by von Lang, but neither Topsøe nor the author has been able to reproduce it in pure ammonium selenate. The author has obtained rhombic mixed crystals of ammonium selenate and sulphate, however, and concludes that ammonium selenate is dimorphous, and that the whole series of sulphates and selenates is probably isodimorphous, as has been shown to be the case with other series of the salts of ammonium, potassium, rubidium, and caesium.

The original crystals investigated by Prof. von Lang have been kindly sent by him to the author, who finds that the majority are truly rhombic, but that they are somewhat impure, containing admixed sulphate, which is probably the reason for their production, for they closely resemble the author's mixed crystals, although much richer in selenate. Besides the rhombic crystals, however, some needles were found of nearly pure ammonium selenate, which are identical with the author's monoclinic form.

The monoclinic crystals of ammonium selenate show a pseudohexagonal primary prism zone very similar to that of the rhombic crystals of the other salts, and the topic parameters calculated on this analogous mode of description show a mean value for the structural dimensions intermediate between the mean value for rubidium and caesium selenates. The molecular volume, refractive indices, dimensions of the optical ellipsoid, and the molecular optical constants are singularly near what would have been expected if the crystals had been truly isomorphically rhombic, and unite in exhibiting ammonium selenate in its proper position in the series immediately after rubidium selenate, precisely analogous to the position of ammonium sulphate in the sulphate series, and which the author has shown is the general position of ammonium crystallographically in the alkali series.

As a side issue, a general explanation has been arrived at for the beautiful optical property of crossed-axial-plane dispersion of the optic axes, ammonium selenate forming the fifth case of this somewhat rare phenomenon which has been met with in the alkali series.

*107. *The Vapour Pressures of Binary Mixtures. Part I. The Possible Types of Vapour Pressure Curves.* By ARTHUR MARSHALL.

The curves showing the variations of partial pressures with molecular composition may be classified into four types, of which three relate to substances which are miscible in all proportions, and are distinguished one from the other according as p/x , the partial pressure divided by the molecular proportion, is less than, equal to, or greater than the vapour pressure of the pure substance. The corresponding total pressure curves are obtained by adding together the two partial pressure curves, which are of the same type. By differentiating the equation of Duhem and Margules, $x \log p_1 + (1-x) \log p_2 = 0$, it has been found possible to classify the total pressure curves into twelve types, all of which are known to occur.

It may be proved that with pairs of substances which are miscible in all proportions only one maximum or minimum can occur in the curve of total pressure. The rule, called by Duhem "Regnault's Law," that in the case of partially miscible liquids the total pressure of the heterogeneous mixture is equal to the vapour pressure of the more volatile constituent in the pure state, has been shown experimentally to be approximately true for methyl acetate and water as well as for ether and water, but it is only a rough approximation and is applicable only to some cases.

The vapour pressures of the following pairs of liquids have been investigated experimentally:—nitroglycerol and acetone, diethylamine and acetone, ethyl alcohol and methyl ethyl ketone, water and methyl ethyl ketone, water and methyl acetate, water and ether, water and amyl alcohol. From the curve of total vapour pressures, the partial pressures may be obtained by a simple graphical method.

*108. *The Behaviour of Acetylene with Electrical Discharges of High Frequency.* By HERBERT JACKSON and DUDLEY NORTHALL LAURIE.

The authors have studied the change, observed by one of them some years ago, when acetylene is subjected to discharges from an ordinary high frequency apparatus. A semi-solid, brown substance is formed, which sets to a hard and very insoluble solid on exposure to air. If care be taken to avoid secondary reactions by too prolonged a passage of the discharge, the composition of the solid agrees with that of acetylene, and is apparently a polymeride. The substance absorbs oxygen readily up to about 8 per cent. When heated out of contact with air, an oil distils and a small quantity of gas is evolved, which consists mainly of methane and hydrogen. The authors are proceeding with the examination of the reactions of the substance, which exhibits many curious properties.

*109. *The Behaviour of the Vapours of Methyl Alcohol and Acetaldehyde with Electrical Discharges of High Frequency.* By HERBERT JACKSON and DUDLEY NORTHALL LAURIE.

The authors have examined the reactions as part of a series of experiments conducted with a view to determining the nature and order of change effected by high-frequency discharges when passed through various gases and vapours. Working with discharges of very short duration, they find the first change in the vapour of methyl alcohol to be the formation of carbon monoxide and hydrogen; in the case of acetaldehyde, the greater part of the vapour breaks up into methane and carbon monoxide, but acetylene and water are also produced simultaneously, though in smaller quantities. Both these changes are reversible, the latter more readily than the former. The authors have continued experiments conducted by one of them some time ago on

a number of saturated and unsaturated compounds, and they have come to the conclusion that in the case of paraffin derivatives the general tendency is for unsaturated bodies to give polymeric varieties under the influence of high-frequency discharges, whilst saturated compounds, under similar conditions, yield substances of simpler structure.

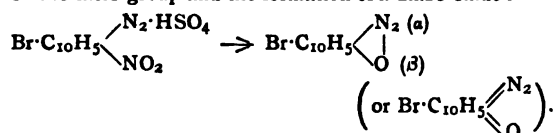
DISCUSSION.

In reply to the President:—Prof. JACKSON said that after trying acetylene made from varying sources, purified acetylene made from calcium carbide and a saturated solution of sodium carbonate was used. The gas was dried over phosphorus pentoxide. No process which could be considered as likely to depolymerise the product gave any acetylene.

In reply to Dr. M. O. Forster:—Prof. JACKSON called attention to the marked activity of the oxidised product obtained after exposing the tacky substance freely to air, and stated that the usual methods for examining for activity in the combined oxygen gave no positive results.

110. "Note on 4-Bromo-2-nitro-1(a)-naphthylamine." By RAPHAEL MELDOLA and HUGH GORDON DALE.

It was shown some years ago by one of the authors and C. H. Desch (*Trans.*, 1892, li., 765) that the above compound could be diazotised under the usual conditions with the formation of diazonium salts which were stable in the presence of excess of sulphuric acid and in cooled solutions. In repeating the diazotisation of the base for another purpose, the authors have found that if the solution of the diazonium sulphate is diluted with water and warmed, an immediate decomposition takes place with the elimination of the nitro-group and the formation of a diazo-oxide:—



This compound is therefore isomeric with the diazo-oxide described by one of the authors and F. W. Streatfield in 1895 (*Trans.*, lxvii., 907) and is identical with the compound more recently described by K. J. P. Orton (*Proc.*, 1902, xvii., 253), with which it agrees in melting point (132°) and other properties.

0.0759 gave 7.3 c.c. moist nitrogen at 11° and 765 m.m. N = 11.46. C₁₀H₇ON₂Br requires N = 11.25 per cent.

The chief point of interest attaching to the present mode of formation of the compound is that it is produced by the elimination of the β-nitro-group, instead of by the displacement of the β-halogen atom, as in Orton's process.

The diazo-oxide on boiling with cuprous chloride solution is converted into 1-chloro-4-bromo-2(β)-naphthol, which crystallises from dilute alcohol in white needles melting at 112° and becoming yellowish-brown on exposure to the air.

0.0851 gave 0.1022 AgCl + AgBr. C₁₀H₆OCIBr requires 0.1026 AgCl + AgBr.

Special interest attaches to this chlorobromonaphthol in connection with the replaceability of the second (β=3) hydrogen atom, from which point of view the compound may be worth detailed study. Attempts to prepare the 1-chloro-4-bromo-2 : 3-naphthaquinone have so far led to negative results, but the experiments will be repeated under different conditions.

111. "Dinitroanisidines and their Products of Diazotisation." (Second communication). By RAPHAEL MELDOLA and FRANK GEORGE C. STEPHENS.

By the nitration of diacetyl-*m*-aminophenol, the authors have obtained a mixture of two isomeric mononitroacetaminophenols, which, on hydrolysis, yield respectively 4-nitro-3-aminophenol (m. p. 185—186°) and 6-nitro-3-aminophenol (m. p. 158°). The nitro-acetaminophenols on nitration both yield the same dinitro-derivative,

4 : 6-dinitro-3-acetaminophenol (m. p. 168°), and this hydrolyses to 4 : 6-dinitro-3-aminophenol. The latter crystallises in dull orange needles of m. p. 231°. The methyl ether (= 4 : 6-dinitro-*m*-anisidine) obtained by direct methylation, or, more advantageously, by the direct nitration of *m*-acetaminophenol methyl ether, crystallises in small, canary-yellow needles of m. p. 208° (acetyl derivative, m. p. 146°). This dinitro-*m*-anisidine does not lose a nitro- or methyl-group on diazotisation, but forms diazonium salts of the ordinary type. From this, combined with former results, the authors conclude that the conditions essential for the elimination of a nitro-group on diazotisation are that there should be a nitro-group, ortho- or para- with respect to the diazonium group, and that this displaceable nitro-group should have a similar group (or possibly a halogen atom) in the ortho-position with respect to itself. Experiments with other dinitro- and haloid-nitroanisidines are in progress.

112. "The Action of Sulphur Dioxide and Aluminium Chloride on Aromatic Compounds." By SAMUEL SMILES and ROBERT LE ROSSIGNOL.

The authors have previously shown (*Trans.*, 1906, lxxxix., 696) that thionyl chloride reacts with phenetole in the presence of aluminium chloride, giving rise successively to a sulphoxide and a sulphonium base; it has since been found that this reaction may be brought about by sulphur dioxide with the aid of the same condensing agent. An examination of the behaviour of various phenolic ethers towards these reagents has shown that the products of the reaction vary according to the nature and position of the substituents in the aromatic nucleus, and in some cases it has been possible to isolate the primary product, the sulphuric acid, in considerable quantity. Preliminary experiments have indicated that the aromatic hydrocarbons are attacked in a similar manner.

113. "Action of Sodium on *aa*-Dichloropropylene." By IDA SMEDLEY.

Van Romburgh and van Dorssen have shown (*Proc. K. Acad. Wetensch. Amsterdam*, 1905, viii., 565) that the simplest hexatriene, CH₂:CH:CH:CH:CH:CH₂, may be synthesised by heating the diformate of *s*-divinyl glycol.

During the past year, the author has endeavoured to prepare a series of hexatrienes from *aa*-dichloropropylene and its homologues. A preliminary account of these experiments is now put forward.

Hübner and Genter state that *aa*-dichloropropylene is indifferent to sodium (*Annalen*, 1860, cxiv., 36). The author finds that if a solution of this substance in petroleum (b. p. 100—110°) is heated with potassium or sodium on a water-bath at about 80° during several days and the petroleum solution separated and fractionally distilled, a small amount of a liquid boiling at 60° and a still smaller quantity of a fraction boiling at about 80° can be isolated. A relatively large proportion of a brown, amorphous solid, insoluble in petroleum or water, is also formed in this reaction.

The fraction boiling at 60° gave the following results on analysis:—

C = 87.0, H = 11.85. C₆H₁₀ requires C = 87.81, H = 12.19 per cent.

Vapour density found 44, calculated 41.

It united readily with bromine, forming a white, solid tetrabromide melting at 51° and containing 80.02 per cent of bromine. The liquid was therefore diallyl.

The amount of the fraction boiling from 80° to 83° was insufficient for identification; it was probably the hexatriene now isolated by van Romburgh and van Dorssen.

The method is being applied for the reduction of *aa*-dichloro-*γ*-phenylpropylene and of *aa*-dichloro-*γ*-methylpropylene; the substances obtained in these experiments are at present under examination.

114. "Resolution of Lactic Acid by Morphine." By JAMES COLQUHOUN IRVINE.

Fermentation lactic acid may be readily resolved into its

active components by the crystallisation of the morphine salts. On neutralising an aqueous solution of the inactive acid with the alkaloid, the sparingly soluble morphine *l*-lactate separated almost quantitatively, whilst the compound with the *d*-acid remained in solution. The crystalline morphine salt was converted into zinc *l*-lactate, which gave $[\alpha]_D^{20} + 6.84^\circ$ and a more accurate test of the purity of the active acid was obtained by its conversion by means of the silver oxide reaction into methyl *l*-methoxypropionate, which showed $[\alpha]_D^{20} + 94.7^\circ$.

The latter compound was reduced by hydriodic acid to *l*-lactic acid, a reaction which shows that silver oxide does not affect the configuration of an active lactate.

By decomposing the syrup containing the soluble morphine *d*-lactate, more than 50 per cent of the theoretical yield of the active zinc salt ($[\alpha]_D^{20} - 6.83^\circ$) was obtained.

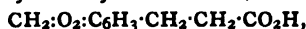
115. "Brazilin and Hæmatoxylin." Part VIII. By WILLIAM HENRY PERKIN, jun., and ROBERT ROBINSON.

The following synthetical experiments have been commenced with the object of obtaining additional evidence as to the constitution of brazilin and hæmatoxylin.

α -Hydrindone, $\text{C}_6\text{H}_4\text{CH}_2\text{CH}_2\text{CO}$, condenses readily with salicylaldehyde in the presence of caustic potash, and is converted into *salical- α -hydrindone*,

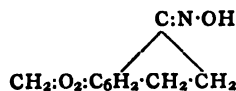
$\text{C}_6\text{H}_4\text{CH}_2\text{C}(\text{CO})\text{CH}(\text{C}_6\text{H}_4)\text{OH}$, which crystallises in yellow needles and decomposes at 206° . It yields a brilliant red potassium derivative, but does not appear to combine with hydrogen chloride.

When methylene dihydrocaffeic acid,—



is treated with phosphorus pentachloride and then with aluminium chloride (compare Kipping, *Trans.*, 1894, lxv., 484), it is converted into *methylene dihydroxy- α -hydrindone*,

$\text{CH}_2\text{O}_2\text{C}_6\text{H}_2\text{CH}_2\text{CH}_2\text{CO}$, which melts at 160° . The *oxime*,—



obtained by the action of hydroxylamine hydrochloride and caustic potash in the usual manner, melts at 246° with decomposition. *isoNitrosomethylenedihydroxy- α -hydrindone*,

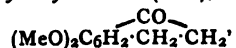
$\text{CH}_2\text{O}_2\text{C}_6\text{H}_2\text{CH}_2\text{C}(\text{CO})\text{N:OH}$, is formed when an alcoholic solution of methylenedihydroxy- α -hydrindone is mixed with *isoamyl* nitrite and hydrochloric acid. It crystallises in yellow needles, decomposes at 230° , and when treated first with phosphorus pentachloride and then with caustic potash, yields *methylenedihydroxyhomophthalic acid*, $\text{CH}_2\text{O}_2\text{C}_6\text{H}_2(\text{CO}_2\text{H})\text{CH}_2\text{CO}_2\text{H}$, which crystallises from water and melts at about 236° .

Benzalmethylenedihydroxy- α -hydrindone,—

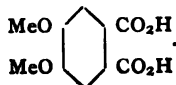


is produced when methylenedihydroxy- α -hydrindone is treated with benzaldehyde and alcoholic potash, and crystallises in pale yellow needles which decompose at about 250° .

4 : 5-Dimethoxy- α -hydrindone (1 : 2),—



is obtained when dimethyldihydrocaffeic acid, $(\text{MeO})_2\text{C}_6\text{H}_3\text{CH}_2\text{CH}_2\text{CO}_2\text{H}$, is treated with phosphorus pentachloride and then with aluminium chloride. It crystallises from benzene, melts at 114° , and, when oxidised with nitric acid, yields *m-hempinic acid*,—

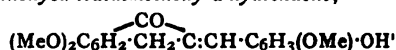


The *isonitroso*-derivative, $(\text{MeO})_2\text{C}_6\text{H}_2\text{CH}_2\text{C}(\text{CO})\text{N:OH}$ crystallises in yellow needles which decompose at about 240° .

Salicaldimethoxy- α -hydrindone,—



is produced when dimethoxy- α -hydrindone is treated, in alcoholic solution, with salicylaldehyde and caustic potash. It crystallises in yellow needles, yields a brick-red hydrochloride, and a brilliant red crystalline potassium derivative. *p-Methoxysalicaldimethoxy- α -hydrindone*,—



is of special interest on account of the close relationship which it bears to trimethylbrazilin. It is obtained when dimethoxy- α -hydrindone is treated with *p*-methoxysalicylaldehyde and caustic potash in alcoholic solution, and crystallises from *isoamyl* alcohol in prisms which decompose at about 230° . It yields a golden-yellow potassium derivative and a crimson hydrochloride.

The authors are engaged in the further investigation of the above and other analogous substances.

116. "A Study of the Reaction between Hydrogen Peroxide and Potassium Persulphate." By JOHN ALBERT NEWTON FRIEND.

It is shown that solutions of hydrogen peroxide and potassium persulphate interact according to the equation $\text{H}_2\text{O}_2 + \text{K}_2\text{S}_2\text{O}_8 = 2\text{KHSO}_4 + \text{O}_2$. The reaction, however, is monomolecular. This is due to the formation of an intermediate and highly unstable compound, directions for the preparation of which are given. Sulphuric acid is found to exert a retarding influence on the reaction, as do the sulphates of sodium, potassium, and manganese, the last-named accelerating slightly as the reaction nears completion. The action of colloidal platinum has also been studied.

117. "The Action of Magnesium Methyl Iodide on Dextrolimonene Nitrosochlorides." By WILLIAM AUGUSTUS TILDEN and FREDERICK GEORGE SHEPHERD.

The α - and β -nitrosochlorides were treated separately, but yielded similar products differing only in melting point and degree of optical activity. The compound formed in each case differs in composition from the nitrosochloride by one atom of oxygen, and therefore has the formula $\text{C}_{20}\text{H}_{32}\text{ON}_2\text{Cl}_2$. It is insoluble in aqueous alkalis and in acids, though easily soluble in the usual organic solvents. Alcoholic potash removes the elements of hydrogen chloride, but the resulting oil rapidly changes in contact with the air. No definite derivatives could be obtained by the use of any reagent except phosphorus pentachloride, which replaces the remaining atom of oxygen by two atoms of chlorine. The product, $\text{C}_{20}\text{H}_{32}\text{N}_2\text{Cl}_4$, is optically active and readily loses two molecules of hydrogen chloride, yielding the compound $\text{C}_{20}\text{H}_{30}\text{N}_2\text{Cl}_2$, which is also optically active and saturated. The product of the action of phosphorus pentachloride does not react as a nitrogen chloride.

118. "Electrolysis of Potassium Ethyl Dipropyl Malonate." By DAVID COWAN CRICHTON.

A concentrated aqueous solution of potassium ethyldipropylmalonate yields on electrolysis the ethyl esters of *α -propyl- β -ethyl-acrylic acid*, *dipropylglycollic acid*, *tetrapropylsuccinic acid*, and probably *dipropylacetic acid*.

The dipropylglycollic acid obtained in the electrolysis was proved to be identical with the "*dipropylloxalic acid*" of Rafalsky.

Tetrapropylsuccinic acid yields an anhydride which is characterised by the same stability towards alkalis as tetraethylsuccinic anhydride.

119. "A New Method for the Measurement of Hydrolysis in Aqueous Solution, based upon the consideration of the Motion of Ions." By ROBERT BECKETT DENISON and BERTRAM DILLON STEELE.

The authors have previously described a method for the direct determination of transport numbers and ionic velocities which necessitates the employment of an auxiliary or indicator solution. This indicator must not be hydrolysed if accurate results are to be obtained, since hydrolysis causes the production of new ions which have an effect on the apparent velocity of the ion which is being subjected to measurement. Thus, if lithium chloride is used as an indicator for potassium chloride, correct numbers for the velocity of the K^+ ion are obtained, since lithium chloride is not hydrolysed in solution. If, however, a salt of the nature of aniline hydrochloride is used as indicator, the H^+ ions which are produced as the result of hydrolysis cause a false value to be obtained for the velocity of the K^+ ion. The difference between the real and apparent velocities of the K^+ ion is dependent on the degree of hydrolysis of the indicator, and it has been found possible, by means of a study of the motion of the different ions in the system, to deduce a formula which gives values for the degree of hydrolysis in the indicator solution well in accordance with the numbers commonly accepted as correct. The only measurements which have to be made are three conductivity determinations. Measurements have been carried out with three salts, namely, the hydrochlorides of aniline, *ortho*- and *para*-toluidine at 18° and 25°. The values obtained for the "hydrolysis constants" are:—

	Aniline hydrochloride.	<i>o</i> -Toluidine hydrochloride.	<i>p</i> -Toluidine hydrochloride.
At 18°	61.9×10^8	46.0×10^8	256×10^8
At 25°	43.6×10^8	29.6×10^8	186×10^8

This gives for the "dissociation constants" of the respective bases (assuming for water $H^+ \times OH^- = 1.2 \times 10^{-14}$) at 25°:—Aniline = 5.2×10^{-10} ; *o*-toluidine = 3.5×10^{-10} ; *p*-toluidine = 2.2×10^{-10} .

From the values obtained for the "hydrolysis constants" of the hydrochlorides at 18° and 25°, the heat of hydrolysis was calculated by the application of van't Hoff's equation. The results were in fair agreement with the values obtained calorimetrically, the numbers being as follows:—Aniline hydrochloride, 8700 cal.; *o*-toluidine hydrochloride, 11,000 cal.; *p*-toluidine hydrochloride, 7900 cal.

120. The Oxidation of Hydrocarbons by Ozone at Low Temperatures." By JULIEN DRUGMAN.

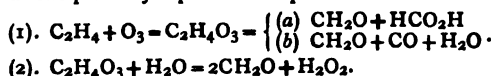
Experimental proof is given that the mode of action of ozone on a saturated and an unsaturated hydrocarbon is very different.

Ozone acts, at the ordinary temperature, very slowly on saturated hydrocarbons such as methane and ethane. The process is one of gradual hydroxylation. In the case of ethane, ethyl alcohol is shown to be the first product formed.

The reaction in the case of an unsaturated hydrocarbon such as ethylene is instantaneous, even at temperatures far below 0°.

A very explosive addition compound is first formed, which decomposes extremely readily, giving oxidation products containing only one carbon atom. The carbon chain is broken at the double bond.

Comparison with the results of Harries' work make it probable that the addition product is an ozonide, but the decomposition of this is more complex than that usually obtained with a liquid or solid ozonide. The following equations probably represent the process:—



Formaldehyde, formic acid, carbon monoxide, hydrogen peroxide, and water are obtained.

121. "Reactions Involving the Addition of Hydrogen Cyanide to Carbon Compounds. Part V. Cyanodihydrocarbons." By ARTHUR LAPWORTH.

The nitrile formed when carvone unites with one molecular proportion of hydrogen cyanide (Hann and

Lapworth, *Proc.*, 1904, xx., 54), is cyanodihydrocarvone, $CHMe \cdot \frac{CO}{CH(CN)} \cdot CH_2 \cdot CH \cdot CMe \cdot CH_2$, and the two acids to which it gives rise on hydrolysis are the corresponding stereoisomeric carboxylic acids. Evidence of the occurrence of reversible dynamic isomerism in the nitrile and the two acids has been obtained.

The properties of these compounds and of a number of their derivatives are described.

122. "Thiocarbamide as a Solvent for Gold." By JAMES MOIR.

Two new complex gold-salts have been obtained by dissolving gold in an acid solution of thiocarbamide (*Proc.*, 1906, xxii., 105). They approximate in composition to $Au_2SO_4 + 6CH_4N_2S$ and $AuCl + 2\frac{1}{2}CH_4N_2S$ respectively, but the author considers that the correct formulæ are $C_6H_{20}N_{12}S_6Au_2(SO_4)$ and $C_8H_{26}N_{16}S_8Au_3Cl_3$ respectively,

and that both contain a complex cation $C_2H_6N_4S_2Au^+$ [most probably $NH \cdot C(NH_2) \cdot S(Au) \cdot NH \cdot CS \cdot NH_2 +$]. The author

suggests that the abnormal cuprous-thiocarbamide salts have similar constitutions.

The chloride has been studied in some detail. It is converted into the compound described by Reynolds, $C_2H_8N_4S_2AuCl$, by boiling with hydrochloric acid. Its formation from aurichloride and thiocarbamide takes place according to the equation:—



On warming with sodium hydrate solution, it gives the olive-green gold compound described by B. Rathke (*Ber.*, 1884, xvii., 307), which the present author formulates as $CH_3N_2S_2Au_3$; the filtrate contains chloride and sulphide of sodium. On heating to 210° until constant, the brown residue appears to have the composition $Au_3S_2 \cdot 3CH_4N_2S$.

Silver nitrate gives a mixture of silver chloride, auric hydrate, and silver thiocarbamide in the proportions required by the formula assigned to the original substance.

Substituted thiocarbamides have no action on gold.

123. "An Improved Beckmann Apparatus for Molecular Weight Determinations." By JAMES MCCONNELL SANDERS.

In the determination of molecular weights by the freezing-point method, employing the Beckmann apparatus as usually constructed, several sources of error have to be contended with, some of which depend on the form of the apparatus, and are considerably augmented when hygroscopic solvents such as acetic acid are used.

One of the chief difficulties is to equalise the overcooling for parallel observations with the solvent and solution, and it is not always easy to add the ice crystals which induce congelation at correspondingly equal temperature intervals from the freezing-point. With the little contrivance ("Impfistif") recommended by Beckmann for this purpose, several errors are liable to be introduced; an unknown, or at least a variable, addition to the weight of the solvent is made, the ice crystals absorb moisture during their transference, and during the time that the side tube is open, moisture is absorbed by the solvent in the tube.

Moreover, when the temperature of the surrounding atmosphere is but a few degrees above the freezing-point of the solvent, it is difficult to ensure the addition of the crystals to the solvent or solution at the precise moment when they are required. Another source of error exists in the introduction of moist atmospheric air by the action of the stirrer.

The author has succeeded in eliminating these errors by the use of the modified form of inner tube described in this paper.

The principal feature of the new inner tube consists in the use of the narrow, almost capillary side tube, A, which is fused into the bottom of the solution tube, and is carried up and bent so as to permit the attachment of a thin rubber tube and small bulb, B. When the solvent has been placed in the containing tube, the height of the column of liquid in A can easily be adjusted by first expelling it com-

pletely by compression of B, then pinching the rubber tube while B is removed, allowed to fill itself with air, and replaced. On releasing the rubber tube a small amount of the solvent rises in A, and the height of the column subsequently increases owing to the contraction of the air in the tube produced by the cooling. The container tube is placed in a wide jacket tube (not shown in the figure) in such a way that the side-tube, A, is in contact with the walls of the latter; a better arrangement is to employ a tube with a lateral canal such as is provided in some hydrometer cylinders.

In operation, the column of solvent in A, being nearer to the source of cold, freezes first, and at the moment desired a mass of crystals can be projected into the main body of the solvent by a sharp compression of the bulb, B.

The pneumatic stirrer, C, is almost self-explanatory; it is an adaptation of the well-known pneumatic "shutter release," found on the majority of photographic cameras. The piston and cylinder are of aluminium or glass ground to fit perfectly (in the author's laboratory use was made of a glass hypodermic syringe of small diameter), the agitator, E, is made by flattening a portion of the length of a piece of platinum wire, and then twisting the flattened portion in the manner shown; the twisted part is coiled into a spiral form, and the other end of the wire fixed to the end of the plunger or piston. The spring which balances the weight of the agitator is made of hard drawn aluminium or platino-iridium wire, sufficient length being given to it to allow of a somewhat extended "stroke."

One of the conveniences attending this arrangement lies in the fact that both stirrer and "crystallisation inducer" can be easily operated without the fatigue attendant on the use of the ordinary form; the operator's hands rest on the table, and he can conveniently observe the thermometer without his attention being distracted by manipulative details.

The ordinary side-tube of the Beckmann apparatus is replaced by the small vertical tube, D, which passes through a third perforation in the rubber stopper. The introduction of the substance can be accomplished by the momentary removal of the stopper of D, or if it should be desired to avoid all possibility of the entrance of atmospheric air, the substance in the form of a compressed tablet can be previously placed in D, and sustained by a bent wire, G, a half revolution of the latter allowing the tablet to drop into the solvent at the desired moment.

The apparatus can easily be constructed by anyone accustomed to glass working, or it can be adapted from a small Soxhlet extraction tube by cutting off the upper and lower parts, and the wide side tube, and bending the narrow syphon tube into the form of A.

(We are indebted to the author for permission to insert the woodcut illustrating the paper).

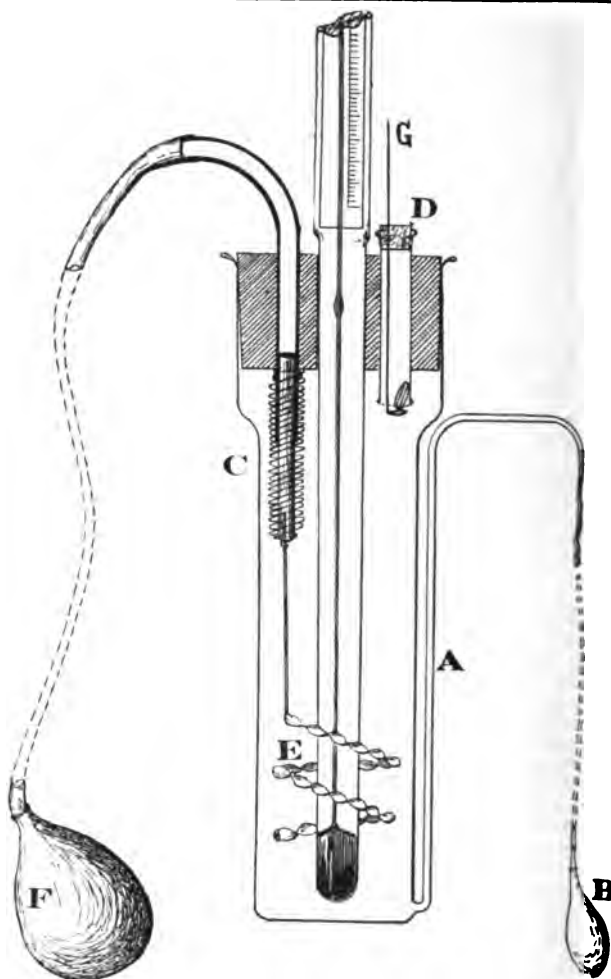
PHYSICAL SOCIETY.

Ordinary Meeting, June 8th, 1906.

Prof. J. PERRY, F.R.S., President, in the Chair.

A PAPER by Mr. H. DAVIES, "On the Solution of Problems in Diffraction by the Aid of Contour Integration," was read by the Secretary.

This paper is an attempt to obtain a solution of diffraction problems by means of contour integration. The method adopted is to obtain a solution for unbounded



space as a contour integral. The special boundary conditions are then accounted for by adding terms to the previous expression. When the complete expression has been obtained it is then evaluated in the form of a series by the aid of Cauchy's Residue Theorem. The special case of a wedge of angle less than 2π and with the light vector parallel to the edge of the wedge is solved in the present case.

Mr. J. GOULD's Experiments with a Vibrating Steel Plate were exhibited by Messrs. Newton and Co.

The phenomena peculiar to this plate may be classified under two heads.—(1) Beats, simultaneously audible and visible; (2) dispersion figures. In addition to these, vortex-vibration, resonance-effects, and many other experiments may be exhibited by using suitable clamps, &c. The dispersion figures are due chiefly to the interaction of two systems of vibrations of the same pitch working at right angles to each other. These two vibration systems may be distinguished as D and L. D is the second of a series of transverse systems in all of which the plate is divided into two equal parts by one nodal line running from end to end, and by one or more nodal lines across the width; in the case where there are two cross lines they are almost identical in position with the two lines proper to the fundamental vibration. But whilst the pitch of the fundamental system is about 70 vibrations per second, that of the D

system is nearly 700. The L system is the first, or lowest, lateral vibration, the direction of vibration being across the width of the plate. These two systems being tuned closely to the same pitch (by means of suitable clamps), careful manipulation with a synchronising rubber will cause any light powder scattered on the plate to be thrown on to the nodal lines proper to D. Strong excitement will, however, immediately bring the L system into activity; the nodal lines will be broken up, and the scattered particles roll to and fro in waves and torrents of great variety, according to the varying distribution of energy between the two vibration systems. Instead of strongly exciting D only, gentle touches of varying intensity may be applied to both systems, according to the effects desired. Dispersion-figures are neither node-figures nor vibration-figures, but, in a sense, they are both, inasmuch as they are perturbations of node lines due to the action of fluctuating vibration. Node-figures are the boundaries of single systems of vibration. Dispersion-figures are resultants of the interaction of at least two systems. Node-figures are fixed; dispersion-figures are fluent, but may be fixed at any stage by stopping the vibration with sufficient promptitude. The principles involved in this phenomenon are identical with those that determine the action of Wilberforce's Spiral Spring. In both cases there is an alternating transfer of energy, by cumulative action, between two synchronising systems of vibration operating at right angles. The principles are identical, but the conditions are very diverse. The spring makes, say, one vibration per second, the plate 700. In the spring action the whole energy is transferred from vertical to horizontal, or *vice versa*, in about 30 vibrations; in the plate action the parallel transfer may require five or six thousand vibrations. With regard to manipulation of the plate, when the interval between the two systems is extremely small, it is almost impossible to excite one without bringing the other into action; in fact, whichever gets the start may be excited through the other. In other words, the manipulation proper for the excitation of one is liable to produce excitation of the other. Beats are most decisive when there is a distinct interval between the two vibration systems; a difference of about 3 or 4 vibrations per second is generally most effective. In this case it is necessary to excite both systems separately so as to obtain vibrations of equal amplitude.

A paper on "Fluid Resistance" was read by Col. R. DE VILLAMIL.

Prof. Hele-Shaw, in a paper on "The Motion of a Perfect Fluid," remarks that one of the most perplexing things in engineering science is the absence of all apparent connection between the higher treatises on hydrodynamics and the vast array of works on practical hydraulics. The reason for this appears to be the immense difference between the flow of an actual liquid and that of a perfect one, owing to the property of viscosity. According to the author this is not the only reason. There appear to be two fundamental difficulties to be got rid of before they can be reconciled. Engineers assume that a liquid can be "pushed" in any rectilinear direction. This, though a very popular notion, is not correct. A liquid, like an electric current, can move in one manner and in one manner only, *viz.*, from a region of higher pressure, or potential, to a region of lower pressure. The author examines the action of a small circular blade moving squarely in a liquid, and shows that, however the matter is considered, the blade cannot "push" the liquid or be pushed by it. The other difficulty is the assumption that in a perfect fluid there can be no resistance of any kind to any moving body moving in it in any velocity. It is only in an infinite ocean of perfect fluid that there would be no resistance. Latterly, however, writers have omitted the words infinite ocean, and have laid down that in all cases a perfect fluid would offer no resistance. This there is no evidence to support.

NOTICES OF BOOKS.

The Life and Experiences of Sir Henry Enfield Roscoe, D.C.L., LL.D., F.R.S. Written by Himself. London: Macmillan and Co., Ltd. New York: The Macmillan Company. 1906.

Those who enjoy biographies and also books with "happy endings," and who can seldom gratify both tastes simultaneously in the perusal of one volume, will be delighted with this account of the life and experiences of Sir Henry Roscoe. The story of his life necessarily includes much of interest about the growth of scientific teaching in England, and the development of Owens College, Manchester, and of the University of London, in connection with which Sir Henry Roscoe did great service to the nation; while the anecdotes of his early student days at Heidelberg and his relationship with Bunsen make lively and, in many cases, amusing reading. His views on the teaching of chemistry, which may be said to show that he belongs in this respect to the older school, are worth careful consideration, as well as his opinions on such subjects as technical education; and the chapters on his work other than scientific force us to recognise that it is indeed seldom that we have the opportunity of reading the life, written by himself, of a man of such many-sided activity and distinguished achievements.

Die Literatur des Vanadins, 1804-1905. By Dr. WILHELM PRANDTL. Hamburg and Leipzig: Leopold Voss. 1906.

THIS collection of publications relating to the chemistry of vanadium and its compounds is a model bibliography, being complete and conveniently arranged. All the more important German and other journals have been carefully searched for the purpose of its preparation, and articles are included in which even isolated and incidental allusions are made to the element or its compounds. Moreover, in all cases translations and abstracts are noticed as well as the originals, so that the compilation must have been a task of considerable magnitude. The arrangement is chronological, and for each article, besides the year of publication and author's name, the full title is given, and, when this does not clearly indicate the scope of the paper, a very brief note pointing out the contents is appended. The book is provided with an index of authors' names, and also a well-classified subject index, which adds greatly to its usefulness.

Flüssige Luft. ("Liquid Air"). By R. NOWICKI and HANS MAYER. Second Edition. Mähr.-Ostau: R. Papauschek. 1906.

THIS small book gives an interesting account of the liquefaction of gases, more especially of air. The early experiments in this direction are simply and clearly explained, while the various processes now usually adopted in France, Germany, and England are described in detail, the text being well illustrated. The most novel and perhaps the most valuable feature of the book is the collection of experiments which can be performed to demonstrate the properties of liquid air; the directions given are very explicit, and those who are contemplating the delivery of a "popular lecture" will find, if they consult this book, many useful hints on manipulation, and the most striking experiments. The second edition has been revised by Hans Mayer, and the most important modern methods of producing liquid gases are described in it, while the new matter given for the first time includes many fresh experiments, besides additional diagrams.

CHEMICAL NOTICES FROM FOREIGN
SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances de l'Académie des Sciences. Vol. cxlii., No. 21, May 21, 1906.

Sulphides, Selenides, and Tellurides of Tin.—H. Pélabon.—The author determines the temperature of fusion of mixtures, in varying proportions, of tin and one of the non-metals—sulphur, selenium, and tellurium. These mixtures, after being raised to a high temperature, are slowly cooled in such a way that the exact temperature when solidification starts can be determined. The results are represented by curves with abscissæ the proportion of non-metal in the mixture (proportion R being expressed in hundredths of the weight of non-metal to the total weight of the mixture) and ordinates the temperature of solidification.

New Methods of Preparation of certain Organic Derivatives of Arsenic.—V. Auger.—The author investigates easy methods of transformation of the principal methyl derivatives of arsenic, starting usually from methylarsinic and cacodylic acids, which are produced commercially at a moderate price. He succeeds in preparing the following compounds:—Methylarsine iodide, CH_3AsI_2 ; methylarsine chloride, CH_3AsCl_2 ; cacodyle chloride, $(\text{CH}_3)_2\text{AsCl}$; cacodyle, $(\text{CH}_3)_2\text{As}.\text{As}(\text{CH}_3)_2$; and tetramethylarsonium, $(\text{CH}_3)_4\text{AsI}$.

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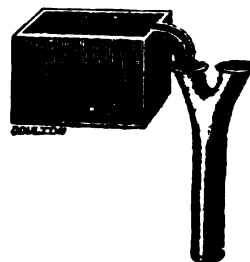
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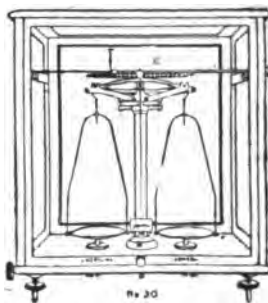
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VOL. XCIII., No. 2431.

THE ESTIMATION OF ORGANIC MATTER WITH PERMANGANATE IN ACID AND IN ALKALINE LIQUIDS.

By C. ALBERTO GARCIA (Instituto de Higiene, Lima, Peru).

THE author comes to the following conclusions:—In the estimation of organic matter with permanganate in an alkaline solution the free ammonia which may exist or be formed in the reagent becomes oxidised and transformed into nitrous or nitric acid, and this increases the expenditure of the permanganate.

Besides the saline ammonia which may be found in the water, the nitrogen of the nitrogenous organic matters liberated in the form of ammonia through the action of the permanganate and of the alkali, whether a bicarbonate or not, increases the expenditure of permanganate, and this explains why larger quantities are obtained in the estimations in an alkaline than in an acid medium when the origin of the organic matters is animal, through the action of the albumenoid ammonia formed.

INVESTIGATIONS ON THE OCCURRENCE OF CHANGES IN THE TOTAL WEIGHT OF SUBSTANCES UNDERGOING CHEMICAL TRANSPOSITIONS.*

(SECOND COMMUNICATION).

By H. LANDOLT.

(Concluded from p. 286).

RESULTS OF THE OBSERVATIONS.

As was explained before, only the final results can be given in this paper, i.e., the alterations in weight obtained in these experiments. These are deduced from the difference of weight of the apparatus A and B—

- I. In a series of weighings before the reaction.
- II. In a series of weighings after the reaction in A.
- III. In a series of weighings after the reaction in B.

Each series consisted of four to eight weighings, and the mean error of the mean of these varied between ± 0.002 and ± 0.008 m.grms. Always denoting the heavier vessel by A, decreases of II. in comparison with I. indicated decrease of weight, and increases showed increases of weight of A. After the reaction in B, weighing III. must again agree approximately with I. Table III. contains also the error in weighing of the result, calculated by summing the mean errors of the two series of weighings.

TABLE III.

Series of weighing.	Difference of weight, A—B.	Mean error.	Result.		
			Vessel.	Alteration of weight.	Error of weighing.
I.	3.588	± 0.003	A	-0.076	± 0.009
II.	3.512	0.006			
III.	3.570	0.005			
			B	-0.058	0.011

I. Determination of the Total Experimental Error by filling the Vessels with Non-reacting Substances.

The two divisions of the reaction vessels were filled either with the same or with two different indifferent liquids (Expts. Nos. 1 to 7). In other cases (Expts. 8 to

19) the apparatus in which a reaction had already taken place was used, and the previous operation was repeated with the then homogeneous mass contained in it. To replace the missing heat of reaction in several experiments (4, 5; 10, 11), the vessel was placed for some time in an air-bath heated to 30–40°. The results are collected in Table IV. These nineteen experiments lead to the following conclusions:—

The alterations in weight given in Col. VII., which include all the errors appearing during an experiment, are both increasing and decreasing, the + sign appearing eight times and the – sign eleven times. The mean of the sum of the two alterations in both directions amounts to $+0.008$ and -0.010 m.grm.

Of the nineteen experiments, seventeen gave an alteration of weight of less than 0.017 m.grm. Only in two cases (Nos. 12 and 13) did this rise to 0.023 and 0.024 m.grm., and this last number may be taken as the maximum error affecting the method. If the limit is moved back a little more, and put at 0.03 m.grm., it is perfectly certain that if an alteration of weight exceeding this amount is observed in an experiment it cannot be caused by errors of observation.

The numbers in Col. VII. include:—

(a) The influences to which the vessels are exposed during the whole operation, and those which may arise through different layers of moisture on the outer glass surface, the slightly unequal volumes of the two vessels, alteration of volume in consequence of the heat of reaction, touching with the transport arrangements, deposit of dust, &c.

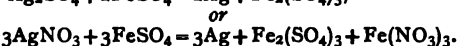
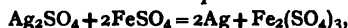
(b) The errors of the balance and the process of weighing. The amount of these is given in Col. VIII., and it is seen to vary between the limits ± 0.006 and ± 0.015 m.grm., thus always remaining much smaller than the total error.

The above maximum error of 0.03 m.grm. relates to the experiments which had been performed since 1901 with the new Rueprecht's balance. As regards the old experiments from the years 1890 to 1892 and 1899, for which Stückrath's and the old Rueprecht's balance were used, and also larger vessels, the mean error of the mean of a series of weighings amounted to ± 0.004 to ± 0.014 m.grm., as the first paper showed. The total experimental error was not determined, but it did not in any case exceed 0.05 m.grm.

Heydeweller (*Drude's Ann. d. Physik.*, 1901, vol. v., p. 404) estimates the greatest error affecting his observations at 0.04 m.grm.

II. Experiments with Substances capable of Reacting.

First Reaction: Silver Sulphate or Nitrate and Ferrous Sulphate.



The two substances were taken in such proportions that the iron vitriol was largely in excess. Nevertheless, as special experiments showed, complete reduction of the two silver salts never took place, but only about 94 to 95 per cent of the total amount of silver separated. When silver sulphate was used, on account of its insolubility, the reduction took place for the most part in the solid state.

The experiments are divided into three groups (Table V.), according to the dates when they were carried out:—

(a) Nos. 1, 2, 3.—Old experiments of the years 1890 and 1892 already described in the first paper. Old Rueprecht's and Stückrath's balance; large β -vessels; weight when filled, about 925 grms.; silver separated, 40 and 60 grms.; error of weighing, up to ± 0.030 m.grm.

(b) Nos. 9, 10, 11.—Experiments of the years 1899 and 1900. Stückrath's balance; large β -vessels weighing about 820 grms.; silver separated, 63.5 grms.; error of weighing, up to ± 0.030 m.grm.

(c) Nos. 4 to 8, 12, 13.—New Rueprecht's balance;

* From *Sitzungsber. d. K. Akademie d. Wissensch.*, 1906, viii., 266.

small η - or O-vessels; weight when filled, 400 to 540 grms.; silver separated, 16.5, 24, 31 grms.; error of weighing, up to ± 0.010 m.grm.

From Table V. it is seen that—

1. The prevailing phenomenon is a decrease of weight, and in nine experiments (Nos. 1 to 6, 9 to 11) this exceeds the maximum error of observation so much that it cannot be explained thus.

2. Generally speaking, as the reaction mass used or the quantity of silver separated increases, the decrease of weight seems to become greater. If the relation of the decrease is calculated to 100 grms. of silver, the following numbers were found for the nine experiments:—

Expt. No.	Decrease of wt. M.grm.	Expt. No.	Decrease of wt. M.grm.
1.	0.42	6.	0.28
2.	0.33	9.	0.31
3.	0.22	10.	0.22
4.	0.27	11.	0.12
5.	0.43		

If but little strict proportionality appears here, nevertheless there is an approximation to proportionality. The mean would be 0.29 m.grm. for each 100 grms. of silver.

3. Experiments Nos. 7, 8, and 12, 13 in which η -vessels were used, the inner glass surfaces of which were covered with a layer of solid paraffin, are an exception. In the first two cases the decrease of weight only slightly exceeds the maximum experimental error of 0.03 m.grm., and in the last two the weight actually remained quite unaltered.

Second Reaction: Iron and Copper Sulphate.



As was mentioned in the introduction, this reaction has been investigated by Heydweiller, using η -tubes, one limb of which was filled with 14 to 18 grms. of iron powder, and the other with an excess of copper vitriol and water. The following differences then appeared:—

(a) When copper sulphate free from acid was used (crystallised from a solution containing some caustic soda) two experiments gave differences of -0.026 and $+0.019$ m.grms., thus no alteration of weight.

(b) If the water used to dissolve the copper vitriol contained a small quantity of alkali, in three experiments the considerable decreases -0.217 , -0.161 , -0.176 m.grm. appeared, which far exceeded the maximum experimental error assumed by Heydweiller, i.e., 0.04 m.grm. Almost equally large alterations, viz., -0.097 and -0.158 m.grm. were obtained on the addition of a small quantity of sulphuric acid to the solution of the copper salt.

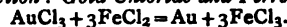
I repeated the experiments, using η -tube of Jena glass, containing:—

- In Experiments 1 and 2—In one limb, 15.0 grms. piano-string wire + 120 grms. pure water; in the other, 70.0 grms. $\text{CuSO}_4 \cdot 5\text{aq}$ + 65 grms. pure water.
- In Experiments 3 and 4—In one limb, 15.0 grms. iron filings + 125 grms. water containing some drops of NaOH ; in the other, 70.0 grms. $\text{CuSO}_4 \cdot 5\text{aq}$ + 70 grms. water containing some drops of NaOH . (67.0 grms. $\text{CuSO}_4 \cdot 5\text{aq}$ stoichiometrically necessary).

With Rueprecht's new balance the results given in Table VI. were obtained. Thus in all four cases decreases of weight occurred, but they were so small in Nos. 1 and 2 that although they exceeded the error in weighing (in the case of No. 2) nevertheless were within the limit of the maximum experimental error of 0.03 m.grm. In agreement with Heydweiller it is thus shown that when copper sulphate free from acid is used no alteration of weight takes place, a result also obtained by Lo Surdo, as I have mentioned. Experiments 3 and 4, when alkali was added, did not give the large increases of weight which Heydweiller observed. In the case of No. 3 it was within the limit of the maximum experimental error of 0.03 m.grm., and in

No. 4 only slightly exceeded it. Thus the occurrence of a decrease of weight cannot be established with certainty. To decide the question, further experiments are necessary, also those with addition of sulphuric acid.

Third Reaction: Gold Chloride and Ferrous Chloride.



η -tubes of Jena apparatus glass were used for the experiment. Into one limb was introduced 122 grms. of a solution of gold chloride prepared from 12.032 grms. of gold, and containing 18.521 grms. of AuCl_3 ; and into the other 122 grms. of ferrous chloride solution, which had been prepared by treating 12.00 grms. of pure iron with hydrochloric acid, and contained 27.20 grms. of ferrous chloride. The quantity stoichiometrically necessary would amount to 23.21 grms. The precipitation of the gold was complete, and during the reaction only a very slight development of heat occurred. (New Rueprecht's balance).

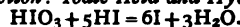
The experiment had to be limited to the vessel A, as B was afterwards damaged.

Date of Experiment: January, 1903.

Gold precipitated (grms.)		12.032
Observed alteration in weight (m.grm.)		-0.009
Error in weighing (m.grm.)		± 0.010

According to this, in this reaction no appreciable alteration of weight occurs. But it must be noticed that the experiment was performed with only small quantities of substances.

Fourth Reaction: Iodic Acid and Hydriodic Acid.



As in the earlier experiments, into the one limb, not aqueous hydriodic acid, but a solution of potassium iodide was introduced, and into the other a sufficient quantity of sulphuric acid besides the iodic acid solution. Thus a premature beginning of the action owing to hydriodic acid gas passing over was avoided. The iodic acid was always used in excess.

Table VII. contains, besides six old experiments, seven new ones. It appears at once from this table that in all thirteen experiments a decrease of weight occurred. In eight cases (Nos. 2, 3, 4, 5, 7, 8, 12, 13) this exceeds the errors of observation (old experiments, 0.05 m.grm; new, 0.03 m.grm.) to a considerable extent; in five cases (Nos. 1, 6, 9, 10, 11), on the other hand, it does not exceed the limits of error.

Fifth Reaction: Iodine and Sodium Sulphite.

This reaction, like the foregoing, may be used for the investigation of the question whether the alterations of weight are connected with the dissociation processes. When iodic acid and hydriodic acid react, iodine being separated, electrons disappear, while in the present case they appear; thus, opposite effects were to be expected. My previous four tests of the reaction had, however, given quite contradictory alterations of weight, both as regards sign and magnitude; thus, the question had not been settled.

Up to the present I have only been able to arrange two new experiments, in which iodine and sodium hydrosulphite (in excess) were allowed to react with one another according to the equation $2\text{I} + \text{NaHSO}_3 + \text{H}_2\text{O} = 2\text{HI} + \text{NaHSO}_4$. In the old experiments iodine and neutral sulphite in the proportion $2\text{I} : \text{Na}_2\text{SO}_3$ were used. All the observations are given in Table VIII. In Experiments 5 and 6 it was not possible to examine apparatus B for alteration in weight, owing to accidents. Of the six experiments, four (Nos. 2, 3, 5, 6) gave a result not exceeding the experimental error, and thus it seems that in this reaction, generally speaking, no appreciable alteration of weight occurs. But it is noticeable that in three cases the alteration was a decrease. As regards Experiments 1 and 4, probably some disturbing influence had come into play.

Sixth Reaction: Uranyl Nitrate and Potassium Hydroxide.
 $2\text{UO}_2(\text{NO}_3)_2 + 6\text{KOH} = \text{K}_2\text{U}_2\text{O}_7 + 4\text{KNO}_3 + 3\text{H}_2\text{O}$.

The experiment was undertaken to see whether when an element of high atomic weight was used a more pronounced alteration of weight would appear. In one limb of the Π -tubes a solution of 63.7 grms. $\text{UO}_2(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (= 50 grms. anhydrous salt) in 96.3 grms. of water was placed, and in the other 25 grms. of caustic alkali (21.35 grms. stoichiometrically necessary), and 135 grms. of water. The result was as follows:—

Date of Experiment: May, 1905.

Kind of Vessel: Π Jena Apparatus Glass.

	No. 1. Apparatus A.	No. 2. Apparatus B.
Potassium uranate precipitated (grms.)	42.28	42.28
Observed alteration in weight (m.grm.)	+0.006	+0.002
Error in weighing (m.grm.)	± 0.009	± 0.010

Thus the weight was perfectly constant. It is to be noted that a very slight development of heat occurred.

Seventh Reaction: Chloral Hydrate and Potassium Hydroxide.
 $\text{C}_2\text{Cl}_3\text{H}_3\text{O}_2 + \text{KOH} = \text{CCl}_3\text{H} + \text{CHKO}_2 + \text{H}_2\text{O}$.

The experiment, already described in the first paper and mentioned here only for completeness sake, had led to the result +0.012 $\pm$ 0.024 m.grm. when 150 grms. of chloral hydrate were converted into 108 grms. of chloroform, and thus no alteration occurred.

Eighth Reaction: Electrolysis of an Aqueous Solution of Cadmium Iodide by an Alternating Current.

The experiment, suggested by my colleague Prof. Nernst, was to show whether, when an element was repeatedly converted from the ordinary condition to a state of ionisation, an alteration of weight could be detected.

The electrolytic apparatus used consisted of a blown glass cylinder, of height 12 c.m. and diameter 4 c.m., closed at the bottom and drawn out to a point at the top. In the interior there were arranged concentrically two circularly bent platinum-foils 9 c.m. high and 2.5 c.m. in diameter, from which platinum wires led outwards through the glass wall. The two surfaces of the electrodes, which were turned towards one another, had areas of 99 and 71 sq. c.m. respectively. Two exactly similar pieces of apparatus were prepared, and one was filled with a concentrated solution of cadmium iodide, containing 40 grms. of salt in 100 c.c. A small quantity of free iodine was purposely added to the solution. When the weight and external volume of the two vessels were compared the following small differences were observed:—

	Weight (grms.)	Volume (c.c.)
Apparatus A	380.158	236.718
" B	380.155	236.702
Difference	0.003	0.016

For the purpose of weighing, the vessels were placed in two equally heavy (86.650 grms.) stands of polished brass, which increased the load on the scale-pan to 466.8 grms.

To produce the current a bipolar direct current motor was used, two points of the coil of which were connected with two sliding rings, from which the alternating current was taken; the number of revolutions was about 1500 per minute. The current intensity was always reduced to 3 ampères.

The process which occurs when the alternating current acts upon a solution of cadmium iodide containing some free iodine is as follows:—The cadmium remains in the liquid as a complex iodide, while a part of the iodine at the two electrodes alternately passes from the ionised condition to the metalloidal and conversely. Possibly a considerable shock to the iodine atom is thus caused. As

a current of 1 ampère in one hour separates 4.025 grm. Ag = 4.731 grms. I, when 3 ampères had acted for forty hours, which were the proportions used in the following experiments, the process must have extended to 567.7 grms. of iodine.

In the first experiments, Apparatus B, when exposed to the alternating current for forty hours, showed a decrease of weight of 0.069 m.grm., and after another forty hours a further decrease of 0.027 m.grm. There was a suspicion, however, that these alterations might be caused by a decrease in the water-film on the outer surface of the vessel, since during the electrolysis the temperature rose to about 30° or 35°. The two vessels were not treated with dilute sulphuric acid after they had been prepared, as was done with the other apparatus, and thus the film might be considerable. The vessels were then laid for a week in 12 per cent sulphuric acid, then for some days in dilute aqueous ammonia, and finally allowed to stand for a longer time under a bell-glass with calcium chloride.

The difference of weight (A - B) of the two vessels had the following values, which are the mean of four separate weighings, after A had been exposed to the action of the alternating current (3 ampères) for forty hours (first for thirty hours, then, after an interruption lasting fourteen hours, for another ten hours).

Date of Experiment: January, 1906.

	A - B.	Mean error in weighing
Before the electrolysis . .	3.145	± 0.004 m.grm.
After the electrolysis . .	3.141	± 0.005 "
Alteration	0.004	± 0.009 "

Thus the alternating current analysis produced no alteration of weight, for the difference 0.004 m.grm. which was obtained, is so small that it falls within the error in weighing. The discussion of this result follows in the last paragraph of the paper.

III. Solution Processes.

Heydweiller found in his experiments that on merely dissolving copper vitriol in water a decrease of weight occurred, especially if small quantities of sulphuric acid were added. The observations in question were given in the earlier table (see Introduction), and from it we see that in five cases decreases amounting to 0.029 to 0.126 m.grm. occurred, and only in one case an increase lying within the limits of error.

My experiments extended to the solution of ammonium chloride, potassium bromide, and uranyl nitrate, when the proportions by weight of salt and water were so adjusted that at the ordinary temperature complete solution of the former resulted. The results, which do not agree well amongst themselves are collected in Table IX. (In this table is also included an old experiment with chloral hydrate, which was described in my first article).

The nine experiments with ammonium chloride show that the solution of this salt is accompanied by no change of weight. Both decreases and increases appeared, which in seven cases exceeded the limit of error of 0.03 m.grm. The increases which were repeatedly obtained, especially the considerable one of Experiment 5, might be caused by the fact that a considerable reinforcement of the water film on the outer glass surface occurred owing to the cooling which the vessel suffered during the process of solution, and perhaps it did not always return to its original state.

The alteration of weight with potassium bromide exceeds the limit of error only very slightly, and with uranyl nitrate and chloral hydrate the weight is perfectly constant.

Copper Sulphate Solution and Alcohol.

As a supplement to the above experiments I examined the case in which the ions of a salt disappear from the solution. In one limb of the Π -tubes 116.5 grms. of absolute alcohol, and in the other 107.8 grms. of copper sul-

phate solution containing 25.0 grms. $\text{CuSO}_4 + 5\text{H}_2\text{O}$ and 8.7 grms. paraffin oil were placed. As a special experiment showed, on mixing these liquids 24.75 grms. $\text{CuSO}_4 + 5\text{H}_2\text{O} = 99$ per cent was separated as a crystalline powder.

Date of Experiment: January-February, 1902.

Kind of Vessel: Π Jena Apparatus Glass.

	No. 1. Vessel A.	No. 2. Vessel B.
$\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ separated (grm.)..	24.75	24.75
Observed alteration of weight (m.grm.)	-0.017	+0.016
Error in weighing	± 0.009	± 0.012

The two experiments show that when a dissociated salt passes into the solid state, no alteration of weight occurs, no more than when a salt is split up into ions.

RESULTS.

1. If we survey all the alterations of weight obtained it strikes us at once that they are chiefly decreases. The

new experiments on the whole show the same result as my earlier ones and those performed by Heydweiller. There were performed—

1. Byme—54 experiments; 42 with —, 12 with + change.
2. By Heydweiller—21 experiments; 19 with —, 2 with + change.

Thus altogether of 75 experiments, which extended to 14 different reactions, 61 corresponding to 81 per cent gave a decrease of weight. In view of the experiments with different substances, when the + and — alterations (8 and 11) obtained were almost equal, this phenomenon cannot be due to errors of observation.

2. The different reactions gave very different decreases. As regards my experiments I observed—

(a) Large decreases in the reactions—

1. Silver sulphate or nitrate, and ferrous sulphate: -0.068 to -0.199 m.grm. in nine experiments.
2. Iodic acid and hydriodic acid: -0.047 to -0.177 m.grm. in nine experiments.

(b) Small alterations, scarcely exceeding the experi-

TABLE IV.

I.	II.	III.	IV.	V.	VI.	VII.	VIII.
Contents of vessels.	Expt. No.	Date of expt.	Kind of vessel.	Weight of contents. Gms.	Vessel.	Observed alteration of weight. M.grm.	Error in weighing. M.grm.
Water	1	Jan., 1903 ..	Π Jena apparatus glass	260	A	-0.003	± 0.012
	2	Feb., " ..	" "	260	A	+0.005	0.015
	3	" " ..	" "	260	B	+0.007	0.014
Water	4	June, 1903 ..	" "	216	A	+0.002	0.011
Paraffin oil	5	" " ..	" "	216	B	-0.007	0.013
Water	6	March, 1903.	Π -tubes quartz glass..	270	A	-0.008	0.009
Mercury	7	" "	" "	270	B	-0.017	0.004
Iodine and sodium sulphate solution . . .	8	Jan.-Feb., '04	Π Jena apparatus glass	350	A	+0.014	0.009
	9	" "	" "	350	B	+0.012	0.008
Copper and ferrous sulphate solution . . .	10	March, 1904.	" "	253	A	+0.015	0.008
	11	" "	" "	253	B	-0.010	0.015
	12	Dec., " "	" "	253	A	-0.023	0.006
	13	" "	" "	253	B	-0.024	0.010
Silver and ferric sulphate solution . . .	14	Feb., 1905 ..	O vessel with vacuum envelope	258	A	-0.009	0.008
	15	" " ..	" "	258	B	+0.006	0.011
	16	March, 1905.	Π Jena apparatus glass with paraffin layer..	300	A	-0.002	0.014
	17	" "	" "	300	B	-0.001	0.015
Uranyl nitrate solution	18	Oct., 1905 ..	Π Jena apparatus glass	367	A	+0.006	—
	19	" " ..	" "	367	B	-0.003	—

TABLE V.

No.	Date of experiment.	Kind of vessel.	Silver separated. Gms.	Reaction in vessel	Observed alteration in weight. M.grm.	Error of weighing. M.grm.
<i>Silver Sulphate and Ferrous Sulphate.</i>						
1.	Oct., 1890	Π -tubes, large, Thuringian glass	40.0	A	-0.167	± 0.021
2.	" "	" "	40.0	B	-0.131	0.030
3.	Jan.-Feb., 1892 ..	" "	60.0	B	-0.130	0.017
4.	Oct.-Nov., 1903 ..	Π -tubes, small Thuringian glass	31.2	A	-0.085	0.009
5.	Jan.-Feb., 1905 ..	O-vessels with vacuum envelope	24.2	A	-0.103	0.009
6.	" "	" "	24.2	B	-0.068	0.010
7.	March, 1905.. ..	Π -tubes, small, J.A.G.* with paraffin layer..	24.2	A	-0.042	0.009
8.	" "	" "	24.2	B	-0.029	0.009
<i>Silver Nitrate and Ferrous Sulphate.</i>						
9.	Oct.-Nov., 1899 ..	Π -tubes, large, Thuringian glass	63.5	A	-0.199	0.030
10.	" "	" "	63.5	B	-0.137	0.028
11.	1900 " "	" "	63.5	A	-0.079	0.013
12.	April-May, 1902 ..	Π -tubes, small, J.A.G. with paraffin layer..	16.5	A	+0.003	0.004
13.	" "	" "	16.5	B	-0.003	0.009

* Jena apparatus glass.

TABLE VI.

No.	Iron and copper sulphate solution.	Date of experiment.	Copper separated. Grms.	Reaction in vessel	Observed alteration of weight. M.grm.	Error in weighing. M.grm.
1.	Without addition of alkali..	Oct.-Nov., 1902 ..	17.0	A	-0.004	± 0.006
2.	" " "	" " "	17.0	B	-0.022	0.007
3.	With addition of alkali ..	Feb.-March, 1904 .	17.0	A	-0.024	0.008
4.	" " "	" " "	17.0	B	-0.041	0.009

TABLE VII.

No.	Date of experiment.	Kind of vessel.	Iodine separated. Grms.	Apparatus.	Observed alteration of weight. M.grm.	Error in weighing. M.grm.
1.	Jan., Feb., March, 1890	∩-tubes, large, old Thuringian glass..	64.9	A	-0.047	± 0.022
2.	" " " "	" " "	64.9	B	-0.114	0.013
3.	Feb.-March, 1891 ..	" " "	80.0	A	-0.103	0.022
4.	" " " "	" " "	80.0	B	-0.102	0.016
5.	May-June, 1891 ..	" " "	160.0	A	-0.177	0.012
6.	" " " "	" " "	160.0	B	-0.011	0.013
7.	Oct., 1901 ..	∩-tubes, small, Jena apparatus glass .	43.3	A	-0.120	0.009
8.	" " " "	" " "	43.3	B	-0.098	0.009
9.	Jan.-Feb., 1904 ..	" " "	64.9	A	-0.004	0.005
10.	Oct., 1904 ..	∩ Quartz glass, closed with resin ..	64.9	A	-0.019	0.004
11.	" " " "	" " "	64.9	B	-0.033	0.014
12.	Dec., 1905 ..	∩-tubes, small, Jena apparatus glass..	54.0	A	-0.083	0.019
13.	" " " "	" " "	54.0	B	-0.053	0.020

Nos. 1-6, old experiments; 7-13, new experiments.

TABLE VIII.

No.	Date of experiment.	Kind of vessel.	Iodine used up. Grms.	Vessel.	Observed alteration in weight. M.grm.	Error in weighing. M.grm.
1.	July-August, 1890 ..	∩-tubes, old Thuringian glass..	90	A	+0.105	± 0.008
2.	" " " "	" " "	90	B	-0.031	0.017
3.	Aug.-Dec., 1891 ..	" " "	110	A	+0.002	0.020
4.	" " " "	" " "	110	B	-0.127	0.017
5.	Oct.-Nov., 1901 ..	∩ Jena apparatus glass ..	50	A	-0.021	0.008
6.	Feb.-March, 1902 ..	" " "	80	A	-0.034	0.009

Nos. 1-4, old experiments; 5 and 6, new experiments.

TABLE IX.

No.	Date of experiment.	Kind of vessel.	Quantities.		Salt in solution. Per cent.	Vessel.	Observed alteration of weight. M.grm.	Error in weighing. M.grm.
			Salt. Grms.	Water. Grms.				
Ammonium Chloride.								
1.	Nov., 1902	∩-tubes, old Thuringian glass ..	37.5	150.0	20	A	-0.024	± 0.019
2.	" " " "	" " " "	37.5	150.0	20	B	-0.002	0.006
3.	May-June, 1902..	O-vessels, with vacuum envelope	23.7	131.6	15.25	A	+0.008	0.006
4.	" " " "	" " " "	23.7	131.6	15.25	B	+0.005	0.009
5.	June, 1902	∩ Jena apparatus glass	44.0	115.4	27.6	A	+0.078 ?	0.009
6.	" " " "	" " " "	44.0	115.4	27.6	B	+0.017	0.010
7.	June-July, 1903..	∩ Quartz vessels closed with resin	60.0	160.0	27.3	A	-0.008	0.008
8.	" " " "	" " " "	60.0	160.0	27.3	B	+0.019	0.014
9.	Nov., 1903	" " " "	51.0	134.0	27.6	B	-0.033	0.010
Potassium Bromide.								
1.	Feb., 1902	∩ Jena apparatus glass	72.5	145.0	33.3	A	-0.038	0.007
Uranyl Nitrate + 6 aq.								
1.	June, 1905	∩ Jena apparatus glass	136.0	136.0	50.0	A	+0.009	0.014
2.	" " " "	" " " "	136.0	136.0	50.0	B	-0.010	0.013
3.	July, 1905	" " " "	136.0	136.0	50.0	B	-0.004	0.017
Chloral Hydrate.								
1.	April, 1891	∩ Thuringian glass	312.0	104.0	75.0	A	-0.003	0.013

mental error of 0.03 m.grm. or even less than this error, in the reactions between—

1. Iron and copper sulphate: -0.004 to -0.041 m.grm.
2. Gold chloride and ferrous chloride: -0.009 m.grm.
3. Iodine and sodium sulphite: -0.021 and -0.034 m.grm. in the two new experiments, while the four old experiments gave + and - results.
4. Uranyl nitrate and potassium hydroxide: $+0.006$ and $+0.002$ m.grm.
5. Chloral hydrate and potassium hydroxide: $+0.012$ m.grm.
6. Ammonium chloride and water: -0.002 to 0.033 m.grm., and $+0.005$ to $+0.019$ m.grm.
7. Potassium bromide and water: -0.038 m.grm.
8. Uranyl nitrate and water: -0.004 , -0.010 , and $+0.009$ m.grm.
9. Chloral hydrate and water: -0.003 m.grm.
10. Copper sulphate solution and alcohol: -0.017 and $+0.016$ m.grm.

Heydweiller had obtained—

(a) Large decreases of weight—

1. In the reaction between iron and acid or alkaline copper sulphate solution: -0.097 to -0.217 m.grm.
2. On dissolving copper vitriol in water containing sulphuric acid: -0.072 to -0.126 m.grm.
3. On mixing copper sulphate solution and caustic potash: -0.045 to -0.092 m.grm.

(b) Small decreases, lying within the limits of experimental error of 0.04 m.grm.—

1. On neutralising acetic acid with ammonia: -0.026 and -0.034 m.grm.
2. In the reaction between barium chloride and sulphuric acid: -0.016 m.grm.
3. If an increase of weight occurred during a reaction, it was always smaller ($+0.002$ to $+0.019$ m.grm.), and lay within the limits of experimental error (0.03 m.grm.). Hence a decrease of weight is the normal phenomenon, and in those cases in which it was only small we cannot with any certainty draw the conclusion that the weight has remained quite constant.
4. No connection has been found between the alteration of weight and the appearance or disappearance of electrons. (See the experiments given in Section III.)
5. There now occurs the question how the decreases of weight are to be explained. It might be suspected that they were due to an external cause, which has not yet been discovered, but taking into account the care with which all the possibilities were investigated, this view seems to be improbable. On the other hand, the fact that the alteration occurs to a marked extent only with certain reactions, such as the reduction of silver or iodine, and is either small or does not occur at all in others, clearly points to a connection with the chemical process.

As the explanation must be of such a nature that it accounts only for decreases and never leads us to expect increases, no other hypothesis seems to remain open, except that already mentioned in the introduction, according to which the phenomenon is due to the releasing of small particles from the chemical atoms. In the case of the radio-active elements, Rutherford and Soddy's hypothesis, which has been thoroughly established, assumes that the cause of the changes they undergo depends upon a gradual disintegration of the atom, which, however, extends only to a small fraction of the total mass, and occurs spontaneously. When chemical reactions take place between two substances, the theory that in consequence of the great shock which the atoms suffer, a small part of their mass splits off, does not seem impossible, especially in view of the considerable decrease of the potential atomic energy which occurs during changes which take place of themselves and with great development of heat. Whether, then, a more complete disintegration of a

few atoms occurs, as with radio-active substances, or whether all the atoms concerned suffer a slight loss, remains undecided. But even in the latter case it would be conceivable that the atoms attacked still retain in essentials their original properties, since they experience only a very small alteration of their composition. Finally, the nature of the released fragments must be left undecided. Electrons do not seem to become free during chemical reactions, at least Martinelli (*Atti R. Acad. dei Lincei*, 1904, [5], xiii., II., 217; *Chem. Centralbl.* 1904, ii., 1096) found that on dissolving copper sulphate in water containing sulphuric acid, or potassium bichromate in water, or on the reduction of silver phosphate with ferrous sulphate, no ionisation of the air surrounding the substances occurred. M. R. Campbell observed the same (*Phil. Mag.*, 1905, [6], ix., 545; *König's, Beibl.*, 1905, 1070).

The theory of the partial disintegration of the atom does not agree with the observation that when a cadmium iodide solution is electrolysed by means of an alternating current, no decrease of weight takes place, although the process of alternately binding and separating off electrons from the iodine atom has extended to about 568 grms. of iodine. Yet it is conceivable that this reaction, although it is connected with a considerable change of the properties of an element, yet causes no such great shock to the iodine atom as the reaction between iodic acid and hydriodic acid, when in the first substance three atoms of oxygen and one of hydrogen, and in the second one atom of hydrogen, are torn from the iodine. Moreover, it is perhaps possible that during the experiment the change of current occurred too rapidly in proportion to the duration of the reaction; a repetition of the experiment with a direct current commuted at suitable intervals would decide this.

Finally, there is the following point to be considered:—The decreases of weight during the reactions will be explicable only if we assume that a part of the mass passes out through the glass wall. The possibility of this is difficult to estimate on account of our ignorance of the particles passing through, but it may be supposed that their magnitude, as they are fragments of an atom, is much less than that of an ordinary molecule. Yet it must be remembered that carbon dioxide slowly passes through glass (Bunsen, *Wied. Ann.*, 1883, xx., 558), and moreover, that the latter, though certainly not until temperatures of about 600° and upwards, is penetrable by hydrogen and by air to a considerable extent (Berthelot, *Comptes Rendus*, 1905, cxl., 817, 821, 1253, and others), just as glowing quartz walls are penetrated by helium (Jaquero and Perrot, *Comptes Rendus*, 1904, cxxix., 789). Moreover, helium, even of the ordinary temperature, seems to penetrate into glass, and to very different degrees (Ramsay and Soddy, *Zeit. Phys. Chem.*, xlviii., 693). On the other hand, glass tubes of diameter 8 m.m. and 1.5 m.m. thickness of wall have been found to be absolutely impermeable towards hydrogen at 40 to 126 atmospheric pressure (Quincke, *Pogg. Ann.*, 1877, clx., 118). If any penetration occurs, the quality of the glass wall must undoubtedly exercise an influence. This is shown by the fact that during the reaction between silver salts and ferrous sulphate there was hardly any decrease of weight when the inner side of the vessel was made impermeable by covering it with a layer of paraffin (Expts. 7, 8, and 12, 13). The thickness of the walls, as well as the composition of the glass, must be taken into account, and special experiments must be performed, in which one and the same reaction will be tested in glass vessels of very different quality. The non-agreement of the results which appeared when the reactions (e.g., iron and copper sulphate) were examined by different workers, is perhaps due to the difference of the glass vessels.

For the present I am obliged to break off the experiments and to publish the results I have so far obtained in this incomplete form. In the future I hope to be able to continue the research at the Physikalisch-Technischen Reichsanstalt, which has kindly offered me suitable accommodation.

THE ATOMIC WEIGHT OF RADIUM AND THE PERIODIC SYSTEM.

By HARRY C. JONES.

THE question of the atomic weight of radium cannot be regarded, as yet, as settled. We have, on the one hand, the determination, by purely chemical means, made by M^{de} Curie, which gave the value 225 (*Ann. Chim. Phys.*, 1904, [7], xxx., 137). On the other, we have the work of Runge and Precht (*Phil. Mag.*, 1903, [6], v., 476), based on a physical method, which gave a much higher value—258.

It is difficult, at present, to decide between these two. The value based upon spectrum analysis (258) places radium in the periodic system after thorium and uranium. In terms of the relation between the mass of the atom and its radio-activity, this is the position which it should occupy. The greater the mass of the atom, the less its stability, and, consequently, the greater its radio-activity.

Various lines of argument have been advanced in favour of the value 225. Perhaps the most important of these is the argument based upon the position of radium in the periodic system. Radium is closely allied chemically to barium, and the value 225 for its atomic weight places it in the same group with barium in series twelve.

This line of argument has had considerable weight, and has been used in favour of the smaller mass for the atom of radium.

The object of this paper is to point out that if the atomic weight of radium is slightly greater than 250, it would find a place in the periodic system quite in accord with all its chemical and physical properties. Indeed, the position which it would then occupy would be rather more satisfactory than that which it would take if its atomic weight were 225.

If the atomic weight of radium were 252 to 256 it would fall in Group II. of the periodic system, along with barium and its homologues, but in a new series—series thirteen.

That this is the case can be seen by a glance at Mendeleeff's table of the periodic system. If we compare the atomic weights of the elements in a given group and in any two succeeding series, we shall find for the elements with the higher atomic weights a difference in the atomic weights, in general, of from 25 to about 31 or 32 units.

Thus, the atomic weight of strontium is 87.6, that of cadmium 112.4—difference 24.8. The atomic weight of barium is 137.4, which differs from that of cadmium by 25 units.

Passing to Group III., the atomic weight of yttrium = 89 differs from that of indium = 115 by 26 units. Indium differs from lanthanum = 138.9 by 23.9 units, lanthanum differs from ytterbium = 173 by 34.1, and ytterbium differs from thallium = 204.1 by 31.1 units.

Turning to Group IV., similar relations manifest themselves. Zirconium, atomic weight 90.6, differs from tin, whose atomic weight is 119.0 by 28.4 units. Tin differs from cerium, whose atomic weight is 140.25, by 21.25 units. Cerium differs from lead, whose atomic weight is 206.9, by 66.65, but since cerium is in Series 9 and lead is in Series 11, and there is no member in this group in Series 10, we must divide the above difference by 2, which gives us a difference per series of 33.32. Lead differs from thorium, whose atomic weight is 232.5, by 25.6 units.

In Group V. the following differences appear:—Vanadium, with an atomic weight of 51.2, differs from arsenic, with an atomic weight of 75, by 23.8. Arsenic differs from niobium = 94 by 19 units. Niobium from antimony = 120.2 by 26.2 points. Antimony from tantalum = 183 by 62.8, but since one series is missing between these two, this difference must be halved = 31.4. Tantalum differs from bismuth = 208.5 by 25.5.

In Group VI. relations of the same kind appear. Chromium, whose atomic weight is 52.1, differs from selenium = 79.2 by 27.1 units. Selenium differs from

molybdenum = 96 by 16.8 units. Molybdenum differs from tellurium = 127.6 by 31.6 points. Tellurium differs from tungsten = 184.0 by 56.4, but as one series is here missing this must be halved = 28.2. Tungsten differs from uranium = 238.5 by 54.5, and halving this, on account of the missing series, we have 27.2.

The above relations suffice to show that two successive members in any group of the periodic system usually differ, in their atomic weights, by a number that is close to from 25 to 28, and may reach 31 or 32, or even higher. This shows that an atomic weight slightly greater than 255, and which might be as high as 257 or 258, would place radium quite as satisfactorily in the periodic system as the lower value 225.

It might be objected that there is no other member of Series 13 known, and that it would be creating a new series just for one element—radium.

It is difficult to see the force of this argument in the light of the facts. In the first place, we know, at present, only two members of Series 12—thorium and uranium. Both of these are radio-active, but only feebly so.

While the placing of radium in Group II., Series 12, brings it in the same group with its chemical relative barium, it at the same time places it in a position which is not in keeping with its most characteristic physical properties. Think of radium as compared with thorium and uranium, or with any other chemical element. It is at least 1,500,000 times as radio-active as uranium. It has the property of charging itself electrically, of lighting itself, and of giving off enormous quantities of heat energy. It is the only element known that has any of these properties to a determinable degree.

Further, it is very unstable, breaking down spontaneously and yielding another chemical element—helium—as one of its decomposition-products.

When we think of these properties alone it seems much more natural to place radium in a series by itself, following the series containing the slightly radio-active elements—thorium and uranium—than to place it in a series with any other chemical element or elements.

If we examine the evidence existing in favour of the value 225 we find, in the first place, the direct determination by M^{de} Curie. It must be admitted that this is far from satisfactory, for a number of reasons. The small quantity of material at disposal made the result less accurate, assuming that the material was pure. A number of the operations to which it was subjected, such as removal of the water by heating in the air, are open to criticism.

Another argument in favour of the 225 value is based upon the fact, now pretty clearly established, that radium is a decomposition-product of uranium, and must have a smaller atomic mass than the substance which produced it. This line of argument has been invalidated by the observation that radium is certainly not formed directly from uranium, but intermediate substances are produced, and, as Joly has pointed out (*Nature*, 1904, lxx., 80), the radium probably results from the interaction of these products with some of the elements in pitchblende. Its atom could then easily have a mass greater than the atom of uranium itself, since radium would thus be a product of synthesis and not of decomposition. It must be stated that certain determinations by Watts (*Phil. Mag.*, July, 1903, [6], vi.) and others, using relations between the spectral lines, led also to a value close to 225. The meaning of this remains to be explained.

The direct argument in favour of the higher value is furnished by the beautiful investigation of Runge and Precht. By means of certain relations between the spectral lines and the atomic weights of the elements producing them, which have been shown to hold for the members of a given group, as the magnesium, calcium, strontium, barium group, they were led to an atomic weight for radium of 257.8, or in round numbers 258.

As shown above, this would not move radium "two rows further down in the column Mg, Ca, Sr, Ba," as stated by Runge and Precht, and to which objection must be

offered, but *only one row*, making it the only member of Series 13.

The atomic weight of radium can be settled, finally, only after a considerable amount of pure material is available. As some time must elapse before this is realised, the best we can do at present is to take the evidence available. The object of the present note, as has been stated, is to call attention to the fact that the argument based upon the periodic system, in favour of 225 as the atomic weight of radium, is not well founded. Indeed, the evidence from the periodic system is certainly as strong, and in the opinion of the writer much stronger, in favour of a higher value, which would be very nearly that found by Runge and Precht.—*American Chemical Journal*, xxxiv., No. 4.

PERKIN MEMORIAL AND INTERNATIONAL JUBILEE OF THE COAL-TAR COLOUR INDUSTRY.

THE year 1906 marks the Fiftieth Anniversary of the epoch-making discovery by William Henry Perkin of the dyestuff "Mauve" by which the foundation was laid of the coal-tar colour industry, and a great stimulus given to the study of organic chemistry.

The discovery, which was destined to have such far-reaching consequences, was made in the Easter vacation of 1856, whilst Perkin, then only a lad of eighteen, was working in a private laboratory he had fitted up in his father's house. The colouring matter was patented on August 26th of the same year, and in the early part of the following year the erection of the first coal-tar colour works was commenced at Greenford Green, near Harrow. Here mauve was soon produced in quantity, and here were manufactured later other coal-tar dyestuffs, including artificial alizarin. From these small beginnings the industry has now grown to dimensions which neither the discoverer nor any man of his time could have foreseen. Not only has an enormous and highly scientific industry been established, the value of which with its collateral branches has been estimated at over twenty million pounds per annum, but the dyeing and textile industries, important and flourishing trades in this country, have been subjected to a complete revolution by which they are being emancipated from their former rule-of-thumb methods and changed from empirical arts to branches of applied science. A profound influence has also been exerted upon the entire chemical trade of the world, and several daughter industries, such as the manufacture of synthetical medicinal agents, antiseptics, synthetic perfumes, artificial sweetening materials, &c., have been called into existence. Side by side and closely interrelated with this great technical progress is the immense stimulus which the establishment and rapid growth of the coal-tar industry has given to the study of pure organic chemistry, especially that of ring carbon compounds.

The discoverer, Dr. Perkin, being fortunately still with us in full activity, it has been widely felt both in this country and abroad that a fitting celebration of the jubilee of the industry should be held which should be made the occasion of a public tribute to its founder in recognition of his great services to chemical industry and chemical science.

At a public meeting held at the the Mansion House, on February 26th, under the chairmanship of the Lord Mayor, the following resolutions were unanimously adopted:—

- I. That in view of this being the Fiftieth Anniversary of the foundation of the coal-tar colour industry, it is desirable that steps should be taken to commemorate the event, and to do honour to W. H. Perkin, the founder.
- II. That an appeal be made in this country and abroad for subscriptions for carrying out the following objects:—

1. The presentation to Dr. Perkin for his lifetime of an oil portrait of himself executed by an eminent artist, the portrait to become the property of the nation at his death.
2. The execution of a marble bust of Dr. Perkin to be placed in the rooms of the Chemical Society.
3. The establishment of a "Perkin Research Fund" for the promotion of chemical research, to be administered through the Chemical Society.

In carrying out these recommendations the hearty support has been secured of the entire scientific world, and a strong international committee has been formed comprising the most prominent men of science and industry in this and other countries.

The subscriptions already received amount to over £2000.

In inviting co-operation it is pointed out that donations may be allocated specifically, if desired, to any one of the above mentioned objects.

The following is the complete programme which has been arranged for the Jubilee Celebration to take place in London on July 26th and 27th:—

July 26th.—

- 11.0 a.m. Meeting at the Royal Institution for the Presentation to Dr. Perkin of Portrait, Bust, Addresses, &c. Ladies are invited.
- 7.0 p.m. Banquet at the Whitehall Rooms, Hotel Metropole. Many distinguished guests are expected to be present.

July 27th.—

- 2.0—6.0 p.m. Visit to the original Works at Greenford Green where Mauve was first manufactured, and Garden Party at Dr. Perkin's house. Train from Paddington (G.W.Ry.) at 2.15 p.m. to Greenford. Return from Sudbury by G.C.Ry. at 6.0 p.m. Ladies are invited.
- 8.30 p.m. Soirée at the Leathersellers' Hall, at the invitation of Dr. and Mrs. Perkin. Ladies are invited.

Institute of Chemistry.—An examination in Biological Chemistry will be held at the Laboratories of the Institute on Tuesday, October 23rd, 1906, and three following days. This examination is open to any Fellow or Associate of the Institute, to any Candidate whose application for admission to the Final Examination has been accepted by the Council, and to any Candidate who has passed the Intermediate Examination of the Institute. The Examination extends over at least four days, and may be theoretical, practical, written, and oral. The syllabus includes Biological Chemistry, with special reference to Fermentation, Enzyme-action, the Chemistry and Bacteriology of Food-stuffs, Water Supply, and Sewage Disposal, and to the application of Biological Chemistry to Industries and manufactures. Candidates intending to enter for this Examination are recommended to study the following subjects:—1. Elementary Biology. 2. The Morphology, Physiology, and Life History of Bacteria, Yeasts, and Moulds, in their relation to Food, Water Supply, the Treatment of Sewage, Agriculture, and the Fermentation Industries. (A special study of pathogenic organisms is not demanded, but the Candidate should acquire a knowledge of such as are of importance in relation to Food and to Water Supply). Practical work should include:—(a) General bacteriological methods and preparation of pure cultures; (b) Microscopy, the staining and mounting of preparations, and the recognition of species; (c) Fermentation changes caused by micro-organisms. 3. Enzymes and their actions. 4. The methods employed in the examination and estimation of the carbohydrates. 5. The proteids and their decomposition products.

PROCEEDINGS OF SOCIETIES.

THE ROYAL SOCIETY.

CONVERSAZIONE AT BURLINGTON HOUSE.

THE second, the Ladies' Soirée, took place on June 20th. Among the more important exhibits were the following:—

Sir JAMES DEWAR, F.R.S.

1. New Charcoal Calorimeter and Thermoscope.—Charcoal at the temperature of liquid hydrogen that has absorbed at atmospheric pressure considerable quantities of helium or hydrogen—or alternatively of nitrogen, oxygen, or air at their respective boiling points—is utilised in this instrument as a material that, by reason of changes in the volume of the occluded gas, exhibits great sensibility to heat and light radiation, and can be used in calorimetry at the temperature of solid hydrogen.

2. Charcoal Vacua.—Electric Discharge Tubes showing gradual gas absorption by charcoal cooled in liquid air until, after the Röntgen radiation stage, the electric resistance becomes so great that a discharge will not pass.

3. Spectrum Tubes.—(1). The less condensable gases of the atmosphere—Helium and Neon. (2). The more condensable gases of the atmosphere—Krypton and Xenon. Each set of gases being separated by the charcoal method.

4. Some Scientific Uses of Liquid Air.—(1). Electric ice crystals. (2). Rough measures of relative thermal conductivities in metals and alloys, by observing the height of the deposited ice cap when similar wires are placed along side each other and the ends immersed in liquid air. The relative conductivities are as the squares of the height of the ice columns. (3). Spheroidal state of liquid air on the surface of different fluids and solutions, showing changes of volatility from the varying amount of vapour condensation; at the same time exhibiting interesting rotatory and translatory movements.

Mr. OLIVER S. DAWSON.—Photographic Prints in Natural Colours (Smith-Merckens process).

The specimens of prints shown have been made in an ordinary printing frame, by one exposure to light, and when taken out of the frame possess the colours which they exhibit. Ordinary "printing-out" paper is only capable of recording black and white, and the intermediate shades of grey. This "printing-out" paper records every colour and shade. It necessitates using in the printing frame, instead of the ordinary negative, a coloured positive. The cost of colouring a positive from an ordinary negative is small.

ORDNANCE COLLEGE, WOOLWICH.—Microphotographs of Brass, &c.

Two of the photos are of the same place on the piece of metal. One of them has been simply rotated on its axis through a few degrees. The same side illumination in each case. The dark structures in the one show as light ones in the other.

Mr. J. E. STEAD, F.R.S.—A Triple Alloy of Tin—Antimony—Arsenic, polished and etched, showing bright curved crystals embedded in a soft matrix or eutectic. (See p. 231).

Mr. KENNETH J. TARRANT.—Photographs of Electrical Discharges, at atmospheric pressure and in vacuo.

A series of photographs showing the characteristic discharge from an interrupted continuous current and a high frequency oscillating one, also between parallel conductors both insulated and bare, of varying capacity, and with various condenser conditions. Also the apparently spiral (vortex) nature of the discharge both in air and in vacuo.

Mr. R. G. DURRANT.—Evidence to show that Ionic Separation occurs when Solutions of Acids or of Salts are allowed to Diffuse into Sensitised Jellies or Solutions.

Tubes, diagrams, and experiments to illustrate that—(1). Acids when they diffuse into blue litmus jellies produce a purple zone in advance of the diffusion-front and (where the acid has a bleaching anion) a yellow-red zone behind the diffusion-front. (2). An acid mixed with litmus, on diffusing, leaves the litmus behind. (3). Hydrogen occluded by palladium would appear to be partially acidic. (4). When concentrated silver nitrate solution diffuses into purple litmus under suitable circumstances, red, blue, colourless and grey-blue—corresponding to H, OH, NO₃, and Ag appear in this order. At certain periods sharp bands may form whose boundaries, if measured from the diffusion start, almost exactly correspond to the known mobilities of these ions.

Messrs. CARL ZEISS, Jena.—Photomicrographic Apparatus for Ultra-violet Light. (Designed by Dr. A. Köhler).

The apparatus consists of a table-top with spark stand, collimator, prism of quartz (which separates rays of different wave-lengths proceeding from the source of light), a collector (which combines the rays of each wave-length into a spark image), a vertical camera with time shutter, microscope with sole-plate; the whole on a second table-top. The camera is mounted on a graduated rod so arranged that it can be swung aside, a special searcher with carrier is provided to be interchanged with camera. The objectives are monochromats specially computed by Dr. M. von Rohr, member of the scientific staff of Messrs. Carl Zeiss, Jena. The objectives are made of fused quartz corrected for $\lambda = 275 \mu\mu$, and 160 m.m. tube length. The highest power is of 1.25 N.A., which when working with ultra-violet light is equal in resolving power to an ideal objective of 2.50 N.A. when white light is used. The lenses of eyepieces and condenser are made of quartz. The rays of the required wave-length on emerging from the collector fall upon a reflecting prism underneath the stand which directs them into the microscope. The image is projected upon a fluorescent screen in the searcher and examined by means of a powerful magnifier. The searcher is so constructed that when the camera is turned in its place the image will appear sharp on the ground-glass screen when a camera extension of about 30 c.m. is used. The source of light is supplied by a current of sparks of Leyden jars charged by an induction coil for greater convenience. Photographs of diatoms taken with this apparatus are shown on the table.

Dr. O. SILBERRAD and Dr. R. C. FARMER.—Stability Test for Cordite.

Illustrating a method recently devised at the Chemical Research Department, Royal Arsenal, Woolwich, for the determination of the stability of cordite and other propellant explosives. It is well known that these explosives decompose gradually on storage, and may eventually ignite spontaneously, if their stability be not tested from time to time. The principle of the new test is based upon the results of several thousand experiments, and is the only method known which gives reliable results with cordite. The test has been adopted by the Service, and will shortly be made use of as a safeguard against spontaneous explosions in powder magazines, particularly in the tropics, where the deterioration takes place most rapidly. In examining cordites the procedure is briefly as follows:—50 grms. of the explosive are maintained at 70°C. in a glass vessel fitted with a mercury manometer: the alteration in pressure is measured at intervals. A contraction takes place at first owing to the absorption of oxygen from the air; subsequently a gradual expansion occurs, the former of these phenomena has never previously been observed.

Sir WILLIAM CROOKES, F.R.S.

1. Stereoscopic Photographs, taken by Sir W. Crookes on the occasion of the visit of the British Association to South Africa in the autumn of 1905.

2. Occurrence of the Diamond.—"Blue Ground" in which diamonds are found; from the 2120 ft. Level, De Beers Mine. Diamantiferous gravel from the Pulsator, De Beers Mine. Selected stones from the Pulsator, De

Beers Mine. Cut and polished section of a piece of silicified wood, found by Mr. Jesse Browne about twelve years ago in the untouched "Blue Ground" of the Du Toits Pan Diamond Mine, Kimberley; the wood bears a great resemblance to the wood of the Camel-Tree (*Acacia Giraffa*) of South Africa. Polished section of the Canyon el Diablo Meteorite, in which diamonds have been found.

Models of Crystals of Diamond.—(1). Diamond in the form of a hexakisoctahedron (the forty-eight scalenohedron), or a solid figure contained by forty-eight scalene triangles. According to Professor Maskelyne this occurs as a self-existent form only in the diamond. (2). Diamond in the form of a hexakisoctahedron and octahedron; from Sudafrika. (3). Diamond in the form of octahedron with intersections. (4). Diamond from Brazil. (5). Diamond from Sudafrika. (6). Diamond from Brazil. (7). A macle or twin crystal, showing its formation from an octahedron with curved edges.

Mr. G. F. HERBERT SMITH.—Precious Stones and Simple Methods for their Identification.

This exhibit illustrates the variety of precious stones available for ornamental purposes. A gem stone must be hard enough to resist the abrasive action of ordinary dust, and at the same time be either transparent or, if opaque, of pleasing colour. The number of mineral species suitable for the purpose is not so restricted as popularly supposed. The names employed by jewellers frequently differ considerably from the scientific nomenclature, being often associated with certain colours rather than particular species, e.g.—topaz (yellow), sapphire (blue), ruby (red), emerald (green), and amethyst (violet). The colour though the most obvious character of a stone is the least trustworthy; and the hardness while of immense importance as regards its durability is of little discriminative value. On the other hand, the optical characters (refractivity, double refraction, and dichroism) and the specific gravity may be easily and accurately determined, and lead to the precise identification of the stone. In the case of practically all faceted transparent stones the refractivity and double refraction are sufficient for the purpose, and the stone need not be removed from its setting. Some simple apparatus for the identification of precious stones is exhibited.

Dr. G. T. MOODY.—Specimens Illustrating the Indifference of Oxygen towards Iron in presence of Water and the Effect of the Admission of Carbonic Acid.

(1). Specimen of soft Swedish iron which has been in contact with air and water both completely freed from carbonic acid for five weeks. During this period approximately 56 litres of air passed over the iron, exposing the metal to a volume of oxygen exceeding thirty times the amount required to completely convert it into ferric oxide. (2). Specimen of the same iron which had remained perfectly bright after exposure for three weeks to water and 32 litres of air, freed from carbonic acid. At the end of the period named, air cleaned by its passage through a tower packed with moist pumice-stone, but containing its normal amount of carbonic acid, was admitted. After 72 hours, during which time approximately 16 litres of air passed through, and when considerable rusting had occurred, the tube was sealed.

Dr. F. D. CHATTAWAY.—Copper Mirrors obtained by the Deposition of Metallic Copper upon Glass.

The method of silvering glass by depositing the metal in a thin film by reduction of some soluble silver compound has long been employed in the production of mirrors, but, hitherto, no method of similarly depositing copper in a brilliant film has been discovered. The exhibit shows a number of glass vessels on which copper has been thus deposited by a slow reduction of the black oxide. The metal being protected from the air such mirrors retain their lustre permanently.

Prof. W. GOWLAND.

1. Portion of a Meteorite containing Diamonds found

near Cañon Diablo, Arizona, and specimens of Diamonds extracted from it.

2. Alloys of Copper and Calcium.—A series of alloys ranging from 0.8 to 61.5 per cent of calcium. All are brittle, and those containing 6 to 7 per cent calcium extremely hard. The higher alloys decompose water, and are readily oxidised in the air. Specimens are also exhibited showing the effects of calcium on lead, tin, bismuth, aluminium, and coinage bronze.

Mr. A. A. C. SWINTON.—Visibly Luminous Electrical Discharges in vacuo obtained with comparatively low Electrical Pressures.

Edison, Fleming, and others have shown that the passage of the electric discharge in vacuo is much facilitated by heating the cathode. Owen and Wehnelt have proved that this effect is enormously increased if the heated cathode be coated with oxides of the alkaline metals. The present experiments show that similar results can be obtained by coating the cathode with radium, and that the effect will be greater when the cathode is heated than obtains without heating.

In the Meeting Room Sir WILLIAM CROOKES gave a Demonstration of Lantern Slides and Experiments in illustration of some Properties of the Diamond.

Lantern Slides.—Surface workings at Wesselson Mine. Diagrammatic section of the "Pipe." The "Pipe," Kimberley Mine. Heap of diamonds in the De Beers Sorting Office; 25,000 carats. Diamonds sorted into sizes. Large diamonds. Large crystal of diamond. Curious crystals of diamond. The Cullinan diamond. Triangular markings on diamond (natural and artificial). Glass shavings of spiral form. Radio active diamonds. Diamond from meteorite. Artificial diamond from granulated iron. Artificial diamond from explosion vessel.

Experiments.—A. Sprouting graphite. B. Natural crystal of diamond showing colours in polarised light. C. Natural crystal of diamond in which double refraction is induced by great pressure. D. Hardness of diamond illustrated by squeezing a natural crystal into steel by hydraulic pressure. E. The depolymerisation of diamond in the electric arc.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances de l'Académie des Sciences. Vol. cxlii., No. 22, May 28, 1906.

Variations in the State of Amorphous Carbon under the influence of Temperature and under the Action of Temperature Oscillations.—O. Manville.—When amorphous carbon is reduced to powder and all the occluded gases got rid of by the combined action of heat and low pressure, the author finds that, if the resulting powder is placed in a current of oxygen and the temperature slowly raised, carbonic anhydride and carbon monoxide are produced, the temperature of formation depending on the nature of the carbon, on its physical and chemical state, on the velocity of the current of oxygen, and on the time.

Acid Phosphites of Primary Cyclic Amines.—P. Lemoult.—The author investigates the products of the action of PCl_3 on certain primary cyclic amines. The reactions, which seem very complex, consist in the decomposition by fixation of the elements of water, resulting in the acid phosphites of the amines in question. Hitherto these compounds have not been investigated, on account of the difficulty of their preparation by direct combination of the base with phosphorous acid. The author's preparation is, however, in practice quite simple, and results in the following compounds:—Acid phosphite of aniline, $\text{PO}_3\text{H}_3\cdot\text{C}_6\text{H}_5\text{NH}_2$;

acid phosphite of *o*-toluidine, $\text{PO}_3\text{H}_2\cdot\text{C}_6\text{H}_4\text{CH}_2\text{NH}_2$; and

acid phosphite of *as-m*-xylydine, $\text{PO}_3\text{H}_2\cdot\text{C}_6\text{H}_4\text{CH}_2\text{NH}_2$.

In general, the action of PCl_3 on the primary cyclic amines in presence of ether or chloroform gives phosphorylated products soluble in these solvents, and the hydrolysis of which produces the acid phosphites of the amines used.

Absolute Atomic Weight of Terbium.—G. D. Hinrichs.—M. Urbain's determination of the atomic weight of terbium (159.22) depends on the values $H=1.007$ and $S=32.06$ used in the calculations. The author applies his correction to the ordinary reduction to determine the absolute atomic weight of terbium. The method used is to weigh the crystalline sulphate, $\text{Tb}_2(\text{SO}_4)_3\cdot 8\text{H}_2\text{O}$, and weigh it again after complete desiccation, $\text{Tb}_2(\text{SO}_4)_3$. The author applies his correction, and brings out the absolute atomic weight to be 159.07 as the mean of three experiments.

Pure Ferro-tungstens.—Em. Vigouroux.—By the aluminothermic method, wolfram-irons can be prepared in which the proportion of tungsten reaches 46.25 per cent. These pure ferro-tungstens, when exhausted by dilute hydrochloric acid, which acts upon the free iron only, leave a body in which the proportion of tungsten increases, and is finally maintained at a constant proportion of 68.70 per cent—an amount corresponding to a compound of formula Fe_3W_2 .

Hydro-anthracenic Derivatives.—Marcel Godchot.—The author prepares a series of hydro-anthracenic derivatives by hydrogenation. Octahydroanthranol, $\text{C}_6\text{H}_{10}\text{CH}_2\text{CHOH}$, results from the hydrogenation of hexahydroanthrone. Anthracenehexahydride, $\text{C}_{10}\text{H}_{16}$, is prepared from octahydroanthranol by a process of dehydration. Anthracene tetrahydride by hydrogenation of dihydro-1-oxanthranol by use of H_2 . γ -Dibromoanthracene tetrahydride is prepared from the latter by the action of bromine.

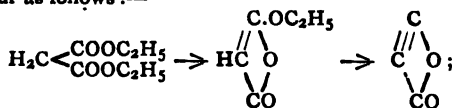
A Qualitative Reaction of Phosphorus.—M. Mauriceau-Beaupré.—The author utilises the property which vapours of phosphoric acid have of corroding fusing glass, and which property is possessed by no other acid except hydrofluoric, as a means of detecting the presence of phosphorus in qualitative reactions.

New Method of Microscopic Analysis of Flour.—G. Gastine.—To detect adulteration of flour the author devises the following method. The flour is impregnated with a colouring solution, slowly dried, and then exposed for some minutes to a temperature of $110-130^\circ$ and examined under the microscope in Canada balsam, when extraneous matter can be detected with ease.

Berichte der Deutschen Chemischen Gesellschaft.
Vol. xxxix., No. 8, 1906.

Hydrates of Chromium Chloride.—A. Werner and A. Gubser.—The following hydrates of chromium chloride are known:— $[\text{Cr}(\text{OH}_2)_6]\text{Cl}_3$, blue hexahydrate; $[\text{Cr}(\text{OH}_2)_5]\text{Cl}$, green hexahydrate; $[\text{Cr}(\text{OH}_2)_4]\text{Cl}_2$, green tetrahydrate; and Godefrey's green decahydrate, prepared by allowing a solution of the green hexahydrate to crystallise at a low temperature. The investigation of this decahydrate showed that two of its chlorine atoms are directly bound with the chromium atom, and one indirectly. All the three green hydrates contain the residue $[\text{Cr}(\text{OH}_2)_4]$. The hydrate with four molecules of water is the normal chloride of this monovalent positive radical, while the decahydrate appears to be $[\text{Cr}(\text{OH}_2)_4]\text{Cl}_2$.

Constitution of Carbon Suboxide.—Arthur Michael.—The structure assumed for the substance obtained when alcohol was split off from malonic acid ester was based upon the theory that the decomposition occurred symmetrically (Diels and Wolf, *Ber.*, 1906, xxxix., 689; *Chem. News*, 1906, xciii., 141). This, however, is in contradiction to our experience of all other such decompositions which lead to the formation of ring derivatives. From analogy with the formation of dimethyl furfuran from acetyl acetone, the decomposition of malonic acid ester might occur as follows:—



and thus the so-called sub-oxide is the lactone of β -hydroxypropionic or propiolic acid. This composition agrees with the chemical behaviour of the substance.

MISCELLANEOUS.

Honours to British Research Laboratories.—Although scientific research receives little encouragement in this country it is gratifying to find that the labours of British scientists are recognised abroad. The Awards to the British Section of the recent Liège Exhibition were distributed at the Mansion House on June 13th, and the following presentations were made:—Wellcome Chemical Research Laboratories, one Grand Prize, one Diploma of Honour, and two Gold Medals; Wellcome Physiological Research Laboratories, one Grand Prize and two Gold Medals. Medals were also awarded to the respective directors of these institutions.

Society of Arts.—The Council of the Society of Arts have awarded the Society's Silver Medal to the following readers of papers during the Session just completed:—Mr. W. F. Mitchell, "The Commerce and Industries of Japan"; Dr. William Arthur Aikin, "Aspects of Voice Development"; Mr. Leon Gaster, A.M.I.E.E., "Progress in Electric Lighting"; Mr. Walter Garstang, M.A., "The Fisheries of the North Sea"; Captain G. S. C. Swinton, "London Traffic"; Mr. Bernard B. Redwood, B.A., "Motor Boats"; Mr. J. B. Millett, "Submarine Signalling"; Professor Thomas Oliver, M.A., M.D., LL.D., "Bridge Building by means of Caissons"; Mr. Clayton Beadle, "Watermarking"; Sir James A. Bourdillon, K.C.S.I., "The Partition of Bengal"; Dr. George A. Grierson, C.I.E., "The Languages of India"; Colonel Sir Arthur Henry McMahon, K.C.I.E., C.S.I., "Seistan"; The Hon. Rodolphe Lemieux, K.C., "French Canada"; The Hon. J. G. Jenkins, "Social Conditions in Australia"; Mr. Louis N. Parker, "Historical Pageants"; Mr. H. Yates Thompson, F.S.A., "Illuminated Manuscripts"; Mr. Harry Powell, "Cut Glass."

MEETINGS FOR THE WEEK.

MONDAY, July 2nd.—Faraday Society. (Special Meeting, to which the public are invited) "Oxidation of Atmospheric Nitrogen in Electric Arcs," by Prof. Kr. Birkeland, of Christiania. Mr. F. W. Harbord will communicate a paper written by Dr. Eugen Haanel, of Ottawa, describing the recent experiments on electric iron and steel smelting that were made at Sault Ste. Marie on behalf of the Canadian Government. (The Meeting will be held at the Society of Arts at 8 p.m.).

THURSDAY, 5th.—Chemical, 8.30. "Saponarin—a New Glucoside, coloured Blue with Iodine," by G. Barger. "Constitution of Umbellulone," by F. Tutin. "Electrolytic Oxidation," by H. D. Law. "Action of Ethyl Iodide and of Propyl Iodide on the Disodium Derivative of Diacetylacetone," by A. W. Bain.

FRIDAY, 6th.—Finsbury Technical College Old Students' Association, 7. (General Meeting).

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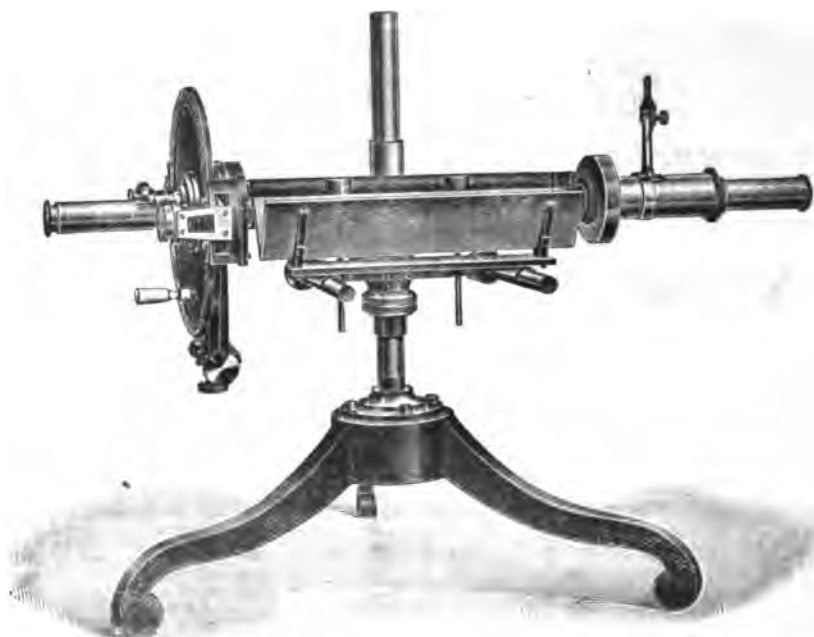
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FACTORY— Reddish, Near STOCKPORT.	CHARLES LOWE & CO. (ESTABLISHED 1860).	TOWN OFFICES— 49, Piccadilly, MANCHESTER.
GOLD MEDAL, PARIS, 1900.	BRONZE MEDAL, PARIS, 1887.	GOLD MEDAL, PARIS, 1878.
MANUFACTURERS OF		
PURE CARBOLIC ACID, Cryst. at 45° C.	Discovered by C. Lowe.	CARBOLIC ACID GLYCERINE SOLUTIONS.
"MEDICINAL" do. Hydrate of		CRESYLIC ACID.
COMMERCIAL do. Cryst. at 35° C.		SULPHO-PHENIC ACID (Cryst.).
" do. No. 1 " 35° C.		SULPHO-PHENATES & SULPHO-CRESYLATES OF SODA, POTASH, ZINC, IRON, and ALUMINA.
" do. No. 2 " 39° C.		
" do. No. 3 " 12° C.		
" do. No. 4 " 0° C.		
CARBOLIC ACID DISINFECTING POWDER.		ACETONE.
		WARDLE'S FERRICUM (Sewage Precipitant).
		PICRIC ACID (Cryst. and Paste).
		AURINE (Rosolic Acid), Cake and Solution.



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